

EXHIBIT E

QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

	<u>Page No.</u>
SECTION I - GENERAL QA/QC PRACTICES	E-2
SECTION II - SPECIFIC QA/QC PROCEDURES	E-3
SECTION III - QUALITY ASSURANCE PLAN	E-5
SECTION IV - STANDARD OPERATING PROCEDURES	E-9
SECTION V - REQUIRED QA/QC OPERATIONS	E-15
SECTION VI - CONTRACT COMPLIANCE SCREENING	E-32
SECTION VII - ANALYTICAL STANDARD REQUIREMENTS	E-33
SECTION VIII - DATA PACKAGE AUDITS	E-38
SECTION IX - PERFORMANCE EVALUATION SAMPLES	E-40
SECTION X - ON-SITE LABORATORY EVALUATIONS	E-43
SECTION XI - DATA MANAGEMENT	E-46

SECTION I

GENERAL QA/QC PRACTICES

Standard laboratory practices for laboratory cleanliness as applied to glassware and apparatus shall be adhered to. Laboratory practices with regard to reagents, solvents, and gases shall also be adhered to. For additional guidelines regarding these general laboratory procedures, see Sections 4 and 5 of the Handbook for Analytical Quality Control in Water and Wastewater Laboratories EPA-600/4-79-019, U.S. EPA Environmental Monitoring Systems Laboratory, Cincinnati, Ohio, September 1982.

SECTION II

SPECIFIC QA/QC PROCEDURES

The quality assurance/quality control (QA/QC) procedures defined herein shall be used by the Contractor when performing the methods specified in Exhibit D. When additional QA/QC procedures are specified in the methods in Exhibit D, the Contractor shall also follow these procedures. NOTE: The cost of performing all QA/QC procedures specified in this Statement of Work are included in the price of performing the bid lot, except for duplicate, spike, and laboratory control sample analyses, which shall be considered separate sample analyses.

The purpose of this document is to provide a uniform set of procedures for the analysis of inorganic constituents of samples, documentation of methods and their performance, and verification of the sample data generated. The program will also assist laboratory personnel in recalling and defending their actions under cross examination if required to present court testimony in enforcement case litigation.

The primary function of the QA/QC program is the definition of procedures for the evaluation and documentation of sampling and analytical methodologies and the reduction and reporting of data. The objective is to provide a uniform basis for sample collection and handling, instrument and methods maintenance, performance evaluation, and analytical data gathering and reporting. Although it is impossible to address all analytical situations in one document, the approach taken here is to define minimum requirements for all major steps relevant to any inorganic analysis. In many instances where methodologies are available, specific quality control procedures are incorporated into the method documentation (Exhibit D). Ideally, samples involved in enforcement actions are analyzed only after the methods have met the minimum performance and documentation requirements described in this document.

The Contractor is required to participate in the Laboratory Audit and Intercomparison Study Program run by USEPA. The Contractor can expect to analyze at least two samples per calendar quarter during the contract period.

The Contractor shall perform and report to SMO and the Technical Project Officer (TPO) as specified in Exhibit B quarterly verification of instrument detection limits (IDL) by the method specified in Exhibit E, by type and model for each instrument used on this contract. All the IDLs shall meet the CRDLs specified in Exhibit C. For ICP methods, the Contractor shall also report, as specified in Exhibit B, linearity range verification, all interelement correction factors, wavelengths used, and integration times.

In this Exhibit, as well as other places within this Statement of Work, the term "analytical sample" is used in discussing the required frequency or placement of certain QA/QC measurements. The term "analytical sample" is defined in the glossary, Exhibit G. As the term is used, analytical sample includes all field samples, including Performance Evaluation samples, received from an external source, but it also includes all required QA/QC samples (matrix spikes, analytical/post-digestion spikes, duplicates, serial dilutions, LCS, ICS, CRDL standards, preparation blanks and linear range analyses) except those directly related to instrument calibration or

calibration verification (calibration standards, ICV/ICB, CCV/CCB). A "frequency of 10%" means once every 10 analytical samples. Note: Calibration verification samples (ICV/CCV) and calibration verification blanks (ICB/CCB) are not counted as analytical samples when determining 10% frequency.

In order for the QA/QC information to reflect the status of the samples analyzed, all samples and their QA/QC analysis shall be analyzed under the same operating and procedural conditions.

If any QC measurement fails to meet contract criteria, the analytical measurement may not be repeated prior to taking the appropriate corrective action as specified in Exhibit E.

The Contractor shall report all QC data in the exact format specified in Exhibits B and H.

Sensitivity, instrumental detection limits (IDLs), precision, linear dynamic range and interference effects shall be established for each analyte on a particular instrument. All reported measurements shall be within the instrumental linear ranges. The analyst shall maintain quality control data confirming instrument performance and analytical results.

In addition, the Contractor shall establish a quality assurance program with the objective of providing sound analytical chemical measurements. This program shall incorporate the quality control procedures, any necessary corrective action, and all documentation required during data collection as well as the quality assessment measures performed by management to ensure acceptable data production.

SECTION III
QUALITY ASSURANCE PLAN

Introduction:

The QAP shall present, in specific terms, the policies, organization, objectives, functional guidelines, and specific QA and QC activities designed to achieve the data quality requirements in this contract. Where applicable, SOPs pertaining to each element shall be included or referenced as part of the QAP. The QAP shall be paginated consecutively in ascending order. Additional information relevant to the preparation of a QAP can be found in Agency and American Society for Testing and Materials publications.

As evidence of such a program, the Contractor shall prepare a written quality assurance plan (QAP) which describes the procedures that are implemented to achieve the following:

- Maintain data integrity, validity, and useability,
- Ensure that analytical measurement systems are maintained in an acceptable state of stability and reproducibility,
- Detect problems through data assessment and establish corrective action procedures which keep the analytical process reliable, and
- Document all aspects of the measurement process in order to provide data which are technically sound and legally defensible.

The QAP shall be available during on-site laboratory evaluation and shall be submitted within 7 days of written request by the APO and/or TPO. The elements of the QAP are listed in the following outline.

A. Organization and Personnel

1. QA Policy and Objectives
2. QA Management
 - a. Organization
 - b. Assignment of QC and QA Responsibilities
 - c. Reporting Relationships
 - d. QA Document Control Procedures
 - e. QA Program Assessment Procedures
3. Personnel
 - a. Resumes
 - b. Education and Experience Pertinent to this Contract

- c. Training Progress
- B. Facilities and Equipment
 - 1. Instrumentation and Backup Alternatives
 - 2. Maintenance Activities and Schedules
- C. Document Control
 - 1. Laboratory Notebook Policy
 - 2. Sample Tracking/Custody Procedures
 - 3. Logbook Maintenance and Archiving Procedures
 - 4. SDG File Organization, Preparation and Review Procedures
 - 5. Procedures for Preparation, Approval, Review, Revision, and Distribution of SOPs
 - 6. Process for Revision of Technical or Documentation Procedures
- D. Analytical Methodology
 - 1. Calibration Procedures and Frequency
 - 2. Sample Preparation Procedures
 - 3. Sample Analysis Procedures
 - 4. Standards Preparation Procedures
 - 5. Decision Processes, Procedures, and Responsibility for Initiation of Corrective Action
- E. Data Generation
 - 1. Data Collection Procedures
 - 2. Data Reduction Procedures
 - 3. Data Validation Procedures
 - 4. Data Reporting and Authorization Procedures
- F. Quality Assurance
 - 1. Data Quality Assurance
 - 2. Systems/Internal Audits
 - 3. Performance/External Audits
 - 4. Corrective Action Procedures

5. Quality Assurance Reporting Procedures
 6. Responsibility Designation
- G. Quality Control
1. Solvent, Reagent and Adsorbent Check Analysis
 2. Reference Material Analysis
 3. Internal Quality Control Checks
 4. Corrective Action and Determination of QC Limit Procedures
 5. Responsibility Designation

Updating and Submitting the QAP:

Initial Submission: During the contract solicitation process, the Contractor is required to submit their QAP to the Administrative Project Officer (APO). Within sixty (60) days after contract award, the Contractor shall maintain on file a revised QAP, fully compliant with the requirements of this contract. The revised QAP will become the official QAP under the contract and may be used during legal proceedings. The Contractor shall maintain the QAP on file at the Contractor's facility for the term of the contract. Both the initial submission and the revised QAP shall be paginated consecutively in ascending order. The revised QAP shall include:

- 1) Changes resulting from A) the Contractor's internal review of their organization, personnel, facility, equipment, policy and procedures and B) the Contractor's implementation of the requirements of the contract; and
- 2) Changes resulting from the Agency's review of the laboratory evaluation sample data, bidder supplied documentation, and recommendations made during the preaward on-site laboratory evaluation.

Subsequent Updates and Submissions: During the term of contract, the Contractor shall amend the QAP when the following circumstances occur:

- 1) The Agency modifies the contract,
- 2) The Agency notifies the Contractor of deficiencies in the QAP document,
- 3) The Agency notifies the Contractor of deficiencies resulting from the Agency's review of the Contractor's performance,
- 4) The Contractor identifies deficiencies resulting from their internal review of their QAP document,
- 5) The Contractor's organization, personnel, facility, equipment, policy or procedures change, or

- 6) The Contractor identifies deficiencies resulting from the internal review of their organization, personnel, facility, equipment, policy or procedures changes.

The Contractor shall amend the QAP within 30 days of when the circumstances listed above result in a discrepancy between what was previously described in the QAP and what is presently occurring at the Contractor's facility.

When the QAP is amended, all changes in the QAP shall be clearly marked (e.g., a bar in the margin indicating where the change is found in the document, or highlighting the change by underlining the change, bold printing the change, or using a different print font). The amended section pages shall have the date on which the changes were implemented. The Contractor shall incorporate all amendments to the current QAP document. The Contractor shall archive all amendments to the QAP document for future reference by the Agency.

The Contractor shall send a copy of the current QAP document within 7 days of a written request by the Administrative Project Officer (APO) and/or Technical Project Officer (TPO) as directed.

Corrective Action:

If a Contractor fails to adhere to the requirements listed in this section, a Contractor may expect, but the Agency is not limited to the following actions: reduction in the numbers of samples sent under this contract, suspension of sample shipment to the Contractor, data package audit, an on-site laboratory evaluation, remedial performance evaluation sample, and/or contract sanctions, such as a Cure Notice.

SECTION IV

STANDARD OPERATING PROCEDURES

Introduction:

In order to obtain reliable results, adherence to prescribed analytical methodology is imperative. In any operation that is performed on a repetitive basis, reproducibility is best accomplished through the use of Standard Operating Procedures (SOPs). As defined by the EPA, an SOP is a written document which provides directions for the step-by-step execution of an operation, analysis, or action which is commonly accepted as the method for performing certain routine or repetitive tasks.

SOPs prepared by the Contractor shall be functional (i.e., clear, comprehensive, up-to-date, and sufficiently detailed to permit duplication of results by qualified analysts). The SOPs shall be paginated consecutively in ascending order.

All SOPs shall reflect activities as they are currently performed in the laboratory. In addition, all SOPs shall be:

- Consistent with current EPA regulations, guidelines, and the CLP contract's requirements.
- Consistent with instruments manufacturers' specific instruction manuals.
- Available to the EPA during an on-site laboratory evaluation. A complete set of SOPs shall be bound together and available for inspection at such evaluations. During on-site laboratory evaluations, laboratory personnel may be asked to demonstrate the application of the SOPs.
- Available to the APO and/or TPO within 7 days of a written request.
- Capable of providing for the development of documentation that is sufficiently complete to record the performance of all tasks required by the protocol.
- Capable of demonstrating the validity of data reported by the Contractor and explain the cause of missing or inconsistent results.
- Capable of describing the corrective measures and feedback mechanism utilized when analytical results do not meet protocol requirements.
- Reviewed regularly and updated as necessary when contract, facility, or Contractor procedural modifications are made.
- Archived for future reference in usability or evidentiary situations.

- Available at specific work stations as appropriate.
- Subject to a document control procedure which precludes the use of outdated or inappropriate SOPs.

SOP Format:

The format for SOPs may vary depending upon the kind of activity for which they are prepared, however, at a minimum, the following sections shall be included:

- Title Page
- Scope and Application
- Definitions
- Procedures
- QC Limits
- Corrective Action Procedures, Including Procedures for Secondary Review of Information Being Generated
- Documentation Description and Example Forms
- Miscellaneous Notes and Precautions
- References

SOPs Required:

The Contractor shall maintain the following SOPs:

1. Evidentiary SOP
Evidentiary SOPs for required chain-of-custody and document control are discussed in Exhibit F.
2. Sample Receipt and Storage
 - a. Sample receipt and identification logbooks
 - b. Refrigerator temperature logbooks
 - c. Security precautions
3. Sample preparation
4. Glassware cleaning
5. Calibration (Balances, etc.)

- a. Procedures
 - b. Frequency requirements
 - c. Preventative maintenance schedule and procedures
 - d. Acceptance criteria and corrective actions
 - e. Logbook maintenance authorization
6. Analytical procedures (for each analytical system)
- a. Instrument performance specifications
 - b. Instrument operating procedures
 - c. Data acquisition system operation
 - d. Procedures when automatic quantitation algorithms are overridden
 - e. QC required parameters
 - f. Analytical run/injection logbooks
 - g. Instrument error and editing flag descriptions and resulting corrective actions
7. Maintenance activities (for each analytical system)
- a. Preventative maintenance schedule and procedures
 - b. Corrective maintenance determinants and procedures
 - c. Maintenance authorization
8. Analytical standards
- a. Standard coding/identification and inventory system
 - b. Standards preparation logbook(s)
 - c. Standard preparation procedures
 - d. Procedures for equivalency/traceability analyses and documentation
 - e. Purity logbook (primary standards and solvents)
 - f. Storage, replacement, and labelling requirements
 - g. QC and corrective action measures

9. Data reduction procedures
 - a. Data processing systems operation
 - b. Outlier identification methods
 - c. Identification of data requiring corrective action
 - d. Procedures for format and/or forms for each operation
10. Documentation policy/procedures
 - a. Laboratory/analyst's notebook policy, including review policy
 - b. Complete SDG File contents
 - c. Complete SDG File organization and assembly procedures, including review policy
 - d. Document inventory procedures, including review policy
11. Data validation/self inspection procedures
 - a. Data flow and chain-of-command for data review
 - b. Procedures for measuring precision and accuracy
 - c. Evaluation parameters for identifying systematic errors
 - d. Procedures to assure that hardcopy and diskette deliverables are complete and compliant with the requirements in SOW Exhibits B and H.
 - e. Procedures to assure that hardcopy deliverables are in agreement with their comparable diskette deliverables.
 - f. Demonstration of internal QA inspection procedure (demonstrated by supervisory sign-off on personal notebooks, internal laboratory evaluation samples, etc.).
 - g. Frequency and type of internal audits (e.g., random, quarterly, spot checks, perceived trouble areas).
 - h. Demonstration of problem identification-corrective actions and resumption of analytical processing. Sequence resulting from internal audit (i.e., QA feedback).
 - i. Documentation of audit reports (internal and external), response, corrective action, etc.
12. Data management and handling
 - a. Procedures for controlling and estimating data entry errors.

- b. Procedures for reviewing changes to data and deliverables and ensuring traceability of updates.
- c. Lifecycle management procedures for testing, modifying and implementing changes to existing computing systems including hardware, software, and documentation or installing new systems.
- d. Database security, backup and archival procedures including recovery from system failures.
- e. System maintenance procedures and response time.
- f. Individuals(s) responsible for system operation, maintenance, data integrity and security.
- g. Specifications for staff training procedures.

Updating and Submitting the SOPs:

Initial Submission: During the contract solicitation process, the Contractor is required to submit their SOPs to the Administrative Project Officer (APO). Within sixty (60) days after contract award, the Contractor shall maintain on file a complete revised set of SOPs, fully compliant with the requirements of this contract. The revised SOPs will become the official SOPs under the contract and may be used during legal proceedings. The Contractor shall maintain the complete set of SOPs on file at the Contractor's facility for the term of the contract. Both the initial submission of SOPs and the revised SOPs shall be paginated consecutively in ascending order. The revised SOPs shall include:

- 1) Changes resulting from A) the Contractor's internal review of their procedures and B) the Contractor's implementation of the requirements of the contract; and
- 2) Changes resulting from the Agency's review of the laboratory evaluation sample data, bidder supplied documentation, and recommendations made during the preaward on-site laboratory evaluation.

Subsequent Updates and Submissions: During the term of contract, the Contractor shall amend the SOPs when the following circumstances occur:

- 1) The Agency modifies the contract,
- 2) The Agency notifies the Contractor of deficiencies in their SOPs documentation,
- 3) The Agency notifies the Contractor of deficiencies resulting from the Agency's review of the Contractor's performance,
- 4) The Contractor's procedures change,
- 5) The Contractor identifies deficiencies resulting from the internal review of their SOPs documentation, or

- 6) The Contractor identifies deficiencies resulting from the internal review of their procedures.

Existing SOPs shall be amended or new SOPs shall be written within 30 days of when the circumstances listed above result in a discrepancy between what was previously described in the SOPs and what is presently occurring at the Contractor's facility. All changes in the SOPs shall be clearly marked (e.g., a bar in the margin indicating where the change is in the document, or highlighting the change by underlining the change, bold printing the change, or using a different print font). The amended/new SOPs shall have the date on which the changes were implemented.

When existing SOPs are amended or new SOPs are written, the Contractor shall document the reasons for the changes, and maintain the amended SOPs or new SOPs on file. Documentation of the reasons for the changes shall be maintained on file with the amended SOPs or new SOPs.

The Contractor shall send a complete set of current SOPs within 7 days of a written request by the Administrative Project Officer and/or Technical Project Officer as directed.

Documentation of the reasons for changes to the SOPs shall also be submitted along with the SOPs. An alternate delivery schedule for submitting the letter and amended/new SOPs may be proposed by the Contractor, but it is the sole decision of the Agency, represented either by the Technical Project Officer or Administrative Project Officer, to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the Technical Project Officer, Administrative Project Officer, and the Contracting Officer why he/she is unable to meet the delivery schedule listed in this section. The Technical Project Officer/Administrative Project Officer will not grant an extension for greater than 30 days for amending/writing new SOPs. The Technical Project Officer/Administrative Project Officer will not grant an extension for greater than 14 days for submission of the letter documenting the reasons for the changes and for submitting amended/new SOPs. The Contractor shall proceed and not assume that an extension will be granted until so notified by the Technical Project Officer and/or the Administrative Project Officer.

Corrective Action:

If a Contractor fails to adhere to the requirements listed in this section, a Contractor may expect, but the Agency is not limited to the following actions: reduction in the number of samples sent under this contract, suspension of sample shipment to the Contractor, data package audit, on-site laboratory evaluation, remedial performance evaluation sample, and/or contract sanctions, such as a Cure Notice.

SECTION V

REQUIRED QA/QC OPERATIONS

This section outlines the minimum QA/QC operations necessary to satisfy the analytical requirements of the contract. The following QA/QC operations shall be performed as described in this Exhibit:

1. Instrument Calibration
2. Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)
3. CRDL Standards for AA (CRA) and ICP (CRI)
4. Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and Preparation Blank (PB) Analyses
5. ICP Interference Check Sample (ICS) Analyses
6. Spike Sample Analysis (S)
7. Duplicate Sample Analysis (D)
8. Laboratory Control Sample (LCS) Analysis
9. ICP Serial Dilution Analysis (L)
10. Instrument Detection Limit (IDL) Determination
11. Interelement Corrections for ICP (ICP)
12. Linear Range Analysis (LRA)
13. Furnace AA QC Analyses

1. Instrument Calibration

Guidelines for instrumental calibration are given in EPA 600/4-79-020 and/or Exhibit D. Instruments shall be calibrated daily or once every 24 hours and each time the instrument is set up. The instrument standardization date and time shall be included in the raw data.

For atomic absorption systems, calibration standards are prepared by diluting the stock metal solutions at the time of analysis. Date and time of preparation and analysis shall be given in the raw data.

Calibration standards shall be prepared fresh daily or each time an analysis is to be made and discarded after use. For atomic absorption systems, prepare a blank and at least three calibration standards in graduated amounts in the appropriate range. One atomic absorption calibration standard shall be at the CRDL. The calibration standards shall be prepared using the same type of acid or combination of acids

and at the same concentration as will result in the samples following sample preparation.

Beginning with the blank, aspirate or inject the standards and record the readings. If the AA instrument configuration prevents the required 4-point calibration, calibrate according to instrument manufacturer's recommendations, and analyze the remaining required standards immediately after calibration. Results for these standards shall be within 5% of the true value. Each standards concentration and the calculations to show that the 5% criterion has been met shall be given in the raw data. If the values do not fall within this range, recalibration is necessary.

The 5% criterion does not apply to the atomic absorption calibration standard at the CRDL.

Calibration standards for AA procedures shall be prepared as described in Exhibit D.

Baseline correction is acceptable as long as it is performed after every sample or after the continuing calibration verification and blank check; resloping is acceptable as long as it is immediately preceded and immediately followed by a compliant CCV and CCB. For cyanide and mercury, follow the calibration procedures outlined in Exhibit D. One cyanide and mercury calibration standard shall be at the CRDL. For ICP systems, calibrate the instrument according to instrument manufacturer's recommended procedures. At least two standards shall be used for ICP calibration. One of the standards shall be a blank.

2. Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)

a. Initial Calibration Verification (ICV)

Immediately after each of the ICP, AA and cyanide systems have been calibrated, the accuracy of the initial calibration shall be verified and documented for every analyte by the analysis of the Initial Calibration Verification Solution(s) at each wavelength used for analysis. When measurements exceed the control limits of Table 1-Initial and Continuing Calibration Verification Control Limits for Inorganic Analyses (in Exhibit E), the analysis shall be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified.

If the Initial Calibration Verification Solution(s) is not available from EPA, or where a certified solution of an analyte is not available from any source, analyses shall be conducted on an independent standard at a concentration other than that used for instrument calibration, but within the calibration range. An independent standard is defined as a standard composed of the analytes from a different source than those used in the standards for the instrument calibration.

For ICP, the Initial Calibration Verification Solution(s) shall be run at each wavelength used for analysis. For CN, the initial

calibration verification standard shall be distilled. This means that an ICV must be distilled with each batch of samples analyzed and that the samples distilled with an ICV must be analyzed with that particular ICV. For aqueous CN samples, the ICV for CN also serves as the Laboratory Control Sample (LCS), and it must be distilled and analyzed as described above. A separate LCS is required for soil CN samples. The values for the initial and subsequent continuing calibration verification shall be recorded on FORM II-IN for ICP, AA, and cyanide analyses, as indicated.

b. Continuing Calibration Verification (CCV)

To ensure calibration accuracy during each analysis run, one of the following standards is to be used for continuing calibration verification and shall be analyzed and reported for every wavelength used for the analysis of each analyte, at a frequency of 10% or every 2 hours during an analysis run, whichever is more frequent. The standard shall also be analyzed and reported for every wavelength used for analysis at the beginning of the run and after the last analytical sample. The analyte concentrations in the continuing calibration standard shall be different than the concentration used for the initial calibration verification and shall be one of the following solutions at or near the mid-range levels of the calibration curve:

1. EPA Solutions
2. NIST Standards
3. A Contractor-prepared standard solution

The same continuing calibration standard shall be used throughout the analysis runs for a Case of samples received.

Each CCV analyzed shall reflect the conditions of analysis of all associated analytical samples (the preceding 10 analytical samples or the preceding analytical samples up to the previous CCV). The duration of analysis, rinses and other related operations that may affect the CCV measured result may not be applied to the CCV to a greater extent than the extent applied to the associated analytical samples. For instance, the difference in time between a CCV analysis and the blank immediately following it as well as the difference in time between the CCV and the analytical sample immediately preceding it may not exceed the lowest difference in time between any two consecutive analytical samples associated with the CCV.

If the deviation of the continuing calibration verification is greater than the control limits specified in Table 1-Initial and Continuing Calibration Verification Control Limits for Inorganic Analyses, the analysis shall be stopped, the problem corrected, the instrument must be recalibrated, the calibration verified and the reanalysis of preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration verification shall be performed for the analytes affected.

Information regarding the continuing verification of calibration shall be recorded on FORM II-IN for ICP, AA and cyanide as indicated.

TABLE 1. INITIAL AND CONTINUING CALIBRATION VERIFICATION
CONTROL LIMITS FOR INORGANIC ANALYSES

Analytical Method	Inorganic Species	% of True Value (EPA Set)	
		Low Limit	High Limit
ICP/AA	Metals	90	110
Cold Vapor AA	Mercury	80	120
Other	Cyanide	85	115

3. CRDL Standards for ICP (CRI) and AA (CRA)

To verify linearity near the CRDL for ICP analysis, the Contractor shall analyze an ICP standard (CRI) at two times the CRDL or two times the IDL, whichever is greater, at the beginning and end of each sample analysis run, immediately preceding the Interference Check Sample (ICS) analyses, but not before the Initial Calibration Verification. In addition, the Contractor shall analyze and report the results for the CRI at a frequency of not greater than 20 analytical samples¹ per analysis run. These analyses of the CRI sample shall be immediately followed by the ICS analyses. (That is, the analytical run sequence shall be CRI, ICSA, ICSAB, CCV and CCB, in that order). This CRI standard shall be run by ICP for every wavelength used for analysis, except those for Al, Ba, Ca, Fe, Mg, Na and K.

To verify linearity near the CRDL for furnace AA, flame AA, and cold vapor AA analyses, the Contractor shall analyze an AA standard (CRA) at the CRDL or the IDL, whichever is greater, at the beginning of each sample analysis run, but not before the Initial Calibration Verification.

Note: Manual and automated cold vapor AA CRA analysis for mercury are required and the results and %R are to be reported on Form II(PART 2)-IN. No specific acceptance criteria have been established by the Agency for the two standards at this time.

4. Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and Preparation Blank (PB) Analyses

a. Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB) Analyses

A calibration blank shall be analyzed at each wavelength used for analysis immediately after every initial and continuing calibration verification, at a frequency of 10% or every 2 hours

¹As defined in Exhibit G, CRI is an analytical sample.

during the run, whichever is more frequent. The blank shall be analyzed at the beginning of the run and after the last analytical sample. Note: A CCB shall be run after the last CCV that was run after the last analytical sample of the run. The results for the calibration blanks shall be recorded on FORM III-IN for ICP, AA and cyanide analyses, as indicated. If the magnitude (absolute value) of the calibration blank result equals or exceeds the IDL, the result shall be reported as specified in Exhibit B. If the absolute value blank result exceeds the CRDL (Exhibit C), terminate the analysis, correct the problem, recalibrate, verify the calibration and reanalyze the preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration blank.

b. Preparation Blank (PB) Analysis

At least one preparation blank (or reagent blank), consisting of deionized, distilled water processed through each sample preparation and analysis procedure (See Exhibit D, Section III), shall be prepared and analyzed with every Sample Delivery Group, or with each batch² of samples digested, whichever is more frequent.

The first batch of samples in an SDG is to be assigned to preparation blank one, the second batch of samples to preparation blank two, etc. (see FORM III-IN). Each data package shall contain the results of all the preparation blank analyses associated with the samples in that SDG.

This blank is to be reported for each SDG and used in all analyses to ascertain whether sample concentrations reflect contamination in the following manner:

- 1) If the absolute value of the concentration of the blank is less than or equal to the Contract Required Detection Limit (Exhibit C), no correction of sample results is performed.
- 2) If any analyte concentration in the blank is above the CRDL the lowest concentration of that analyte in the associated samples shall be greater than or equal to 10x the blank concentration. Otherwise, all samples associated with the blank with the analyte's concentration less than 10x the blank concentration and above the CRDL, shall be redigested and reanalyzed for that analyte (except for an identified aqueous soil field blank). The sample concentration is not to be corrected for the blank value.
- 3) If the concentration of the blank is below the negative CRDL, then all samples reported below 10x CRDL associated with the blank shall be redigested and reanalyzed.

²A group of samples prepared at the same time.

The values for the preparation blank shall be recorded in ug/L for aqueous samples and in mg/Kg for solid samples on FORM III-IN for ICP, AA, and cyanide analyses.

5. ICP Interference Check Sample (ICS) Analysis

To verify interelement and background correction factors, the Contractor shall analyze and report the results for the ICP Interference Check Samples at the beginning and end of each analysis run, but not before the Initial Calibration Verification. In addition, the Contractor shall analyze and report the results for the ICP Interference Check Sample at a frequency of not greater than 20 analytical samples³ per analysis run. These analyses of the Interference Check Samples shall be immediately followed by the analysis of a CCV/CCB pair. The ICP Interference Check Samples shall be obtained from EPA if available and analyzed according to the instructions supplied with the ICS.

The Interference Check Samples consist of two solutions: Solution A and Solution AB. Solution A consists of the interferents, and Solution AB consists of the analytes mixed with the interferents. An ICS analysis consists of analyzing both solutions consecutively (starting with Solution A) for all wavelengths used for each analyte reported by ICP.

The analytical results for those target analytes with CRDLs ≤ 10 ug/L shall fall within $\pm 2x$ CRDL of the analyte's true value (the true value shall be zero unless otherwise stated) in the ICS Solution A (ICSA). For example, if the analysis result(s) for Arsenic (CRDL = 10 ug/L, ICSA true value = 0 ug/L) in the ICSA analysis during the run is ± 19 ug/L, then the analytical result for Arsenic falls within the $\pm 2x$ CRDL window for Arsenic in the ICSA. If the Contractor cannot obtain results that fall within the $\pm 2x$ CRDL window (for analytes with a CRDL ≤ 10 ug/L), then the Contractor shall use an alternate method (e.g., GFAA) to quantitate results for the affected analyte(s) for samples analyzed since the last good ICSA. For the analytes with CRDLs ≤ 10 ug/L, the ICSA results shall be reported from an undiluted sample analysis. Also, the Contractor shall not dilute the Interference Check Samples more than is necessary to meet the linear range values of the instrument.

Results for the ICP analyses of Solution AB during the analytical runs shall fall within the control limit of $\pm 20\%$ of the true value for the analytes included in the Interference Check Samples. If not, terminate the analysis, correct the problem, recalibrate the instrument, and reanalyze the analytical samples analyzed since the last good ICS. This $\pm 20\%$ window does not apply when the IDL exceeds the CRDL for the analytes As, Pb, Se, Tl (see Exhibit C, Table 1, Footnote 1). If true values for analytes contained in the ICS and analyzed by ICP are not supplied with the ICS, the mean shall be determined by initially analyzing the ICS at least five times repetitively for the particular analytes. This mean determination shall be made during an analytical run where the results for the previously supplied EPA ICS met all contract specifications. Additionally, the result of this initial mean

³As defined in Exhibit G, ICSA and ICSAB are analytical samples.

determination is to be used as the true value for the lifetime of that solution (i.e., until the solution is exhausted).

If the ICP Interference Check Sample is not available from EPA, independent ICP Check Samples shall be prepared with interferent and analyte concentrations at the levels specified in Table 2-Interferent and Analyte Elemental Concentrations Used for ICP Interference Check Sample. The mean value and standard deviation shall be established by initially analyzing the Check Samples at least five times repetitively for each parameter on FORM IV-IN. Results shall fall within the control limit of $\pm 20\%$ of the established mean value. The mean and standard deviation shall be reported in the raw data. Results from the Interference Check Sample analyses shall be recorded on FORM IV-IN for all ICP parameters.

TABLE 2. INTERFERENT AND ANALYTE ELEMENTAL CONCENTRATIONS USED FOR ICP INTERFERENCE CHECK SAMPLE

Analytes	(mg/L)	Interferents	(mg/L)
Ag	0.2	Al	500
As	0.1	Ca	500
Ba	0.5	Fe	200
Be	0.5	Mg	500
Cd	1.0		
Co	0.5		
Cr	0.5		
Cu	0.5		
Mn	0.5		
Ni	1.0		
Pb	0.05		
Sb	0.6		
Se	0.05		
Tl	0.1		
V	0.5		
Zn	1.0		

6. Spike Sample Analysis (S)

The spike sample analysis is designed to provide information about the effect of the sample matrix on the digestion and/or measurement methodology. If a digestion is performed, the spike is added before the digestion (i.e., prior to the addition of other reagents) and prior to any distillation steps (i.e., CN-). At least one spike sample analysis (matrix spike) shall be performed on each group of samples of a similar matrix type (i.e., water, soil) and concentration (i.e., low, medium) or for each Sample Delivery Group.⁴

⁴EPA may require additional spike sample analysis, upon Administrative Project Officer request, for which the Contractor will be paid.

If the spike analysis is performed on the same sample that is chosen for the duplicate sample analysis, spike calculations shall be performed using the results of the sample designated as the "original sample" (see section 7, Duplicate Sample Analysis). The average of the duplicate results cannot be used for the purpose of determining percent recovery. Samples identified as field blanks cannot be used for spiked sample analysis. EPA may require that a specific sample be used for the spike sample analysis.

The analyte spike shall be added in the amount given in Table 3-Spiking Levels for Spike Sample Analysis, for each element analyzed. Note: See Table 3 footnotes for concentration levels and applications. If two analytical methods are used to obtain the reported values for the same element within a Sample Delivery Group (i.e., ICP, GFAA), spike samples shall be run by each method used.

If the spike recovery is not at or within the limits of 75-125%, the data of all samples received associated with that spike sample and determined by the same analytical method shall be flagged with the letter "N" on FORMS I-IN and V-IN. An exception to this rule is granted in situations where the sample concentration exceeds the spike concentration by a factor of four or more. In such an event, the data shall be reported unflagged even if the percent recovery does not meet the 75-125% recovery criteria.

For flame AA, ICP, and CN analyses, when the pre-digestion/pre-distillation spike recovery falls outside the control limits and the sample result does not exceed 4x the spike added, a post-digestion/post-distillation spike shall be performed for those elements that do not meet the specified criteria (exception: Ag). Spike the unspiked aliquot of the sample at 2x the indigenous level or 2x CRDL, whichever is greater. Results of the post-digestion/post-distillation spike shall be reported on FORM V(PART 2)-IN. Note: No post digest spike is required for Hg.

In the instance where there is more than one spike sample per matrix and concentration per method per SDG, if one spike sample recovery is not within contract criteria, flag all the samples of the same matrix, level, and method in the SDG. Individual component percent recoveries (%R) are calculated as follows:

$$\% \text{ Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where, SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

When sample concentration is less than the instrument detection limit, use SR = 0 only for purposes of calculating % Recovery. The spike sample results, sample results and % Recovery (positive or negative) shall be reported on FORM V-IN for ICP, AA and cyanide analyses, as indicated.

The units for reporting spike sample results will be identical to those used for reporting sample results in FORM I-IN (i.e., ug/L for aqueous and mg/Kg dry weight basis for solid).

TABLE 3. SPIKING LEVELS FOR SPIKE SAMPLE ANALYSIS

Element	For ICP/AA		For Furnace AA ⁽⁴⁾		Other ⁽¹⁾⁽²⁾
	Water (ug/L)	Soil ⁽²⁾ (mg/Kg)	Water (ug/L)	Soil ⁽²⁾ (mg/Kg)	
Aluminum	2,000	*			
Antimony	500	100	100	20	
Arsenic	2,000	400	40	8	
Barium	2,000	400			
Beryllium	50	10			
Cadmium	50	10	5	1	
Calcium	*	*			
Chromium	200	40			
Cobalt	500	100			
Copper	250	50			
Iron	1,000	*			
Lead	500	100	20	4	
Magnesium	*	*			
Manganese	500	100			
Mercury					1
Nickel	500	100			
Potassium	*	*			
Selenium	2,000	400	10	2	
Silver	50	10			
Sodium	*	*			
Thallium	2,000	400	50	10	
Vanadium	500	100			
Zinc	500	100			
Cyanide					100 ug/L ⁽³⁾

*No spike required. NOTE: Elements without spike levels, and not designated with an asterisk, shall be spiked at appropriate levels.

¹Specified spiking levels are for both water and soil/sediment matrices. Reporting units are ug/L and mg/kg respectively.

²The levels shown indicate concentrations in the final solution of the spiked sample (100 mL for mercury and 200 mL for all other metals) when the wet weight of 1 gram (for ICP, Furnace AA, and Flame AA), or 0.2 grams (for mercury), of sample is taken for analysis. Adjustment shall be made to maintain these spiking levels when the weight of sample taken deviates by more than 10% of these values. Appropriate adjustment shall

be made for microwave digestion procedures where 0.5 grams of sample or 50.0 mL (45.0 mL of sample plus 5.0 mL of acid) of aqueous sample are required for analysis.

³The level shown indicates the cyanide concentration in the final sample solution prepared for analysis (i.e., post-distillation). The final volume of the sample after distillation shall be the basis for the amount of cyanide to be added as the spike. For instance, the full volume distillation procedure will require addition of 25 ug cyanide to the sample prior to distillation (based on the final distillate volume of 250 mL) to meet the specified spiking level; and the midi distillation procedure requires the addition of 5 ug of cyanide to the sample prior to distillation (based on the final distillate volume of 50 mL).

For soil samples, the final sample solution prepared for analysis (i.e., the distillate) must contain cyanide spiked at a concentration of 100 ug/L regardless of the distillation procedure employed or the amount of sample used for distillation. Use the final sample volume after distillation as the basis for the amount of cyanide to add as the spike. The units for reporting soil/solid sample cyanide results shall be mg/kg. To convert from ug/L to mg/kg, use the equation below:

$$\text{mg/kg} = \text{ug/L} \times \frac{\text{final distillate volume (L)}}{\text{sample weight (g)}}$$

⁴If the Contractor uses an Inductively Coupled Plasma (ICP) spectrometer to analyze field samples for those elements (e.g., Arsenic, Lead, Selenium, and/or Thallium) traditionally analyzed by the Graphite Furnace Atomic Absorption (GFAA) spectrometer, the spiking concentrations shown for furnace AA analyses (Table 3, above) shall also apply to the ICP analysis for those elements, provided the ICP IDLs for those elements do not exceed the CRDL. Otherwise, those elements shall be spiked at the ICP levels specified in Table 3. However, before any field samples are analyzed under this contract, the instrument detection limits (in ug/L) shall be determined for each instrument used, within thirty (30) days of the start of contract analyses and at least quarterly (i.e., January, April, July, October), and shall meet the Contract Required Detection Limits (CRDLs) specified in Exhibit C, Page C-1, Table 1. For additional information concerning the instrument detection limit determination see Exhibit E, Section V, item 10 - Instrument Detection Limit (IDL) Determination.

7. Duplicate Sample Analysis (D)

One duplicate sample shall be analyzed from each group of samples of a similar matrix type (i.e., water, soil) and concentration (i.e., low, medium) or for each Sample Delivery Group.⁵ Duplicates cannot be averaged for reporting on FORM I-IN.

⁵EPA may require additional duplicate sample analyses, upon Administrative Project Officer request, for which the Contractor will be paid.

Duplicate sample analyses are required for percent solids. Samples identified as field blanks cannot be used for duplicate sample analysis. EPA may require that a specific sample be used for duplicate sample analysis. If two analytical methods are used to obtain the reported values for the same element for a Sample Delivery Group (i.e., ICP, GFAA), duplicate samples shall be run by each method used. The relative percent differences (RPD) for each component are calculated as follows:

$$RPD = \frac{|S - D|}{(S+D)/2} \times 100$$

Where, RPD = Relative Percent Difference
 S = First Sample Value (original)
 D = Second Sample Value (duplicate)

The results of the duplicate sample analyses shall be reported on FORM VI-IN in ug/L for aqueous samples and mg/Kg dry weight basis for solid original and duplicate samples. A control limit of 20% for RPD shall be used for original and duplicate sample values greater than or equal to 5x CRDL (Exhibit C). A control limit of (\pm) the CRDL shall be used if either the sample or duplicate value is less than 5x CRDL, and the absolute value of the control limit (CRDL) shall be entered in the "Control Limit" column on FORM VI-IN.

If one result is above the 5x CRDL level and the other is below, use the \pm CRDL criteria. If both sample values are less than the IDL, the RPD is not calculated on FORM VI-IN. For solid sample or duplicate results < 5x CRDL, enter the absolute value of the CRDL, corrected for sample weight and percent solids, in the "Control Limit" column. If the duplicate sample results are outside the control limits, flag all the data for samples received associated with that duplicate sample with an "*" on FORMs I-IN and VI-IN. In the instance where there is more than one duplicate sample per SDG, if one duplicate result is not within contract criteria, flag all samples of the same matrix, concentration, and method in the SDG. The percent difference data will be used by EPA to evaluate the long-term precision of the methods for each parameter. Specific control limits for each element will be added to FORM VI-IN at a later date based on these precision results.

8. Laboratory Control Sample (LCS) Analysis

Aqueous and solid Laboratory Control Samples (LCS) shall be analyzed for each analyte using the same sample preparations, analytical methods and QA/QC procedures employed for the EPA samples received. The aqueous LCS solution must be obtained from EPA (if unavailable, the Initial Calibration Verification Solutions may be used). One aqueous LCS must be prepared and analyzed for every group of aqueous samples in a Sample Delivery Group, or for each batch of aqueous samples digested, whichever is more frequent. An aqueous LCS is not required for mercury. For cyanide, a distilled ICV is used as the LCS (see Exhibit E, Section V, item 2).

The EPA-provided solid LCS shall be prepared and analyzed using each of the procedures applied to the solid samples received (exception:

percent solids determination not required). If the EPA solid LCS is unavailable, other EPA Quality Assurance Check samples or other certified materials may be used. One solid LCS shall be prepared and analyzed for every group of solid samples in a Sample Delivery Group, or for each batch of samples digested and/or distilled, whichever is more frequent.

All LCS results and percent recovery (%R) will be reported on FORM VII-IN. If the percent recovery for the aqueous LCS falls outside the control limits of 80-120% (exception: Ag and Sb), the analyses shall be terminated, the problem corrected, and the samples associated with that LCS redigested and reanalyzed.

If the results for the solid LCS fall outside the control limits established by EPA, the analyses shall be terminated, the problem corrected, and the samples associated with that LCS redigested and reanalyzed.

9. ICP Serial Dilution Analysis (L)

Prior to reporting concentration data for the analyte elements, the Contractor shall analyze and report the results of the ICP Serial Dilution Analysis. The ICP Serial Dilution Analysis shall be performed on a sample from each group of samples of a similar matrix type (i.e., water, soil) and concentration (i.e., low, medium) or for each Sample Delivery Group, whichever is more frequent. Samples identified as field blanks cannot be used for Serial Dilution Analysis.

If the analyte concentration is sufficiently high (minimally a factor of 50 above the instrumental detection limit in the original sample), the serial dilution (a five fold dilution) must then agree within 10% of the original determination after correction for dilution. If the dilution analysis for one or more analytes is not at or within 10%, a chemical or physical interference effect must be suspected, and the data for all affected analytes in the samples received associated with that serial dilution shall be flagged with an "E" on FORM IX-IN and FORM I-IN.

The percent differences for each component are calculated as follows:

$$\% \text{ Difference} = \frac{|I - S|}{I} \times 100$$

where, I = Initial Sample Result
 S = Serial Dilution Result (Instrument Reading x 5)

In the instance where there is more than one serial dilution per SDG, if one serial dilution result is not within contract criteria, flag all the samples of the same matrix and concentration in the Sample Delivery Group. Serial dilution results and "E" flags shall be reported on FORM IX-IN.

10. Instrument Detection Limit (IDL) Determination

Before any field samples are analyzed under this contract, the instrument detection limits (in ug/L) shall be determined for each instrument used, within 30 days of the start of contract analyses and at least quarterly (i.e., January, April, July, October), and shall meet the levels specified in Exhibit C.

The Instrument Detection Limits (in ug/L) shall be determined by multiplying by 3, the average of the standard deviations obtained on three nonconsecutive days (e.g., Monday, Wednesday and Friday) from the analysis of a standard solution (each analyte in reagent water) at a concentration 3x-5x the instrument manufacturer's suggested IDL, with seven consecutive measurements per day. Each measurement shall be performed as though it were a separate analytical sample (i.e., each measurement shall be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDLs shall be determined and reported for each wavelength used in the analysis of the samples. In addition, IDLs shall be reported on Form X-IN for each instrument used in reporting results for an SDG and shall be submitted with each data package.

The quarterly determined IDL for an instrument shall always be used as the IDL for that instrument during that quarter. If the instrument is adjusted in any way that may affect the IDL, the IDL for that instrument shall be redetermined and the results submitted for use as the established IDL for that instrument for the remainder of the quarter.

11. Interelement Corrections for ICP

Before any field samples are analyzed under this contract, the ICP interelement correction factors shall be determined prior to the start of contract analyses and at least annually thereafter. Correction factors for spectral interference due to Al, Ca, Fe, and Mg shall be determined for all ICP instruments at all wavelengths used for each analyte reported by ICP. Correction factors for spectral interference due to analytes other than Al, Ca, Fe, and Mg, shall be reported if they were applied.

If the instrument was adjusted in any way that may affect the ICP interelement correction factors, the factors shall be redetermined and the results submitted for use. In addition, all data used for the determination of the interelement correction factors shall be available to the USEPA during an on-site laboratory evaluation. Results from interelement correction factors determination shall be reported on FORM XI(PART 1)-IN, and FORM XI(PART 2)-IN for all ICP parameters.

12. Linear Range Analysis (LRA)

For all ICP analyses, a linear range verification check standard shall be analyzed and reported quarterly (i.e., January, April, July, October) for each element on FORM XII-IN. The standard shall be analyzed during a routine analytical run performed under this contract. The analytically determined concentration of this standard shall be within

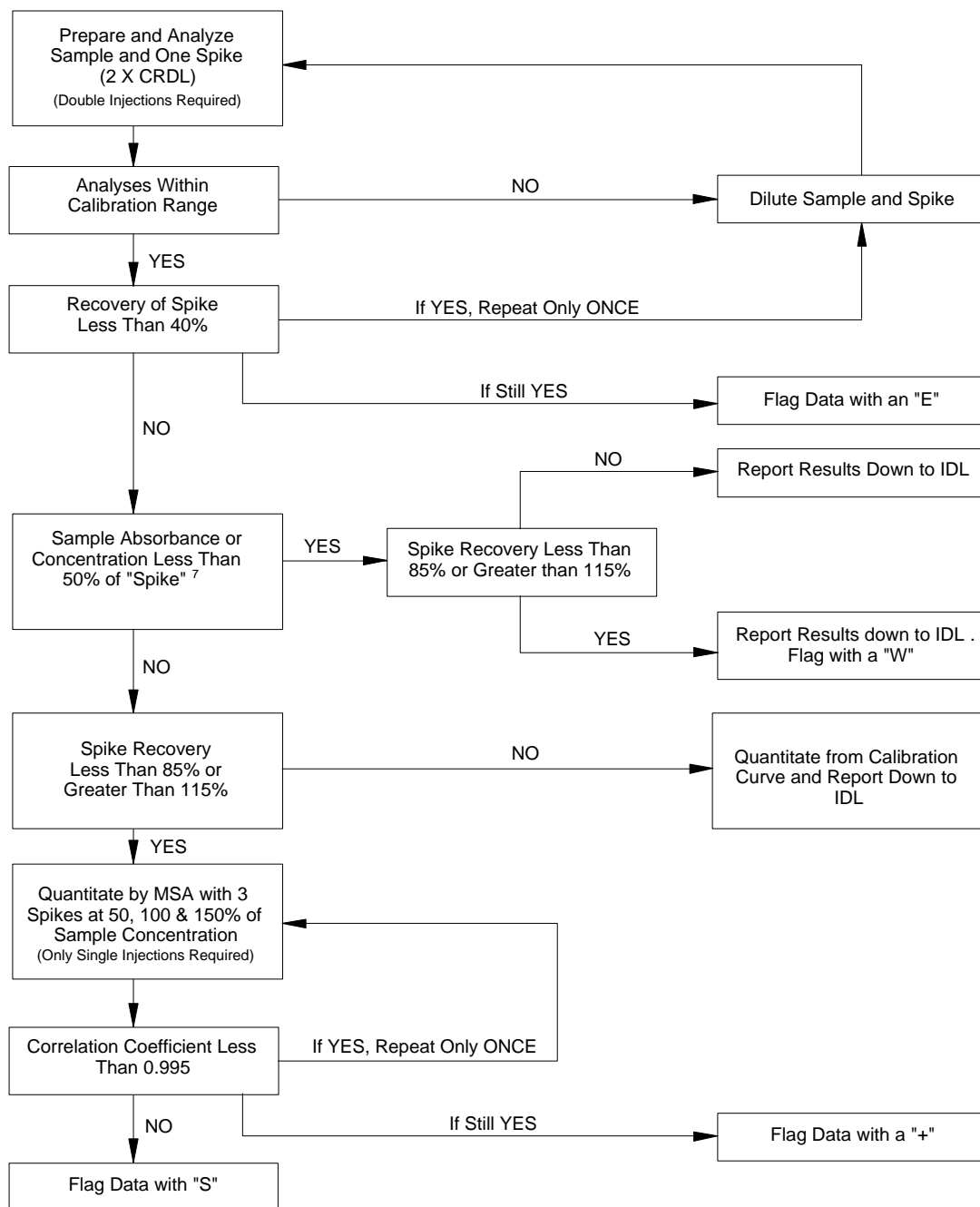
5% of the true value. This concentration is the upper limit of the ICP linear range beyond which results cannot be reported under this contract without dilution of the analytical sample.

13. Furnace Atomic Absorption (AA) QC Analyses

Because of the nature of the Furnace AA technique, the special procedures summarized in Figure 1-Furnace AA Analysis Scheme ("MSA Tree") will be required for quantitation. (These procedures do not replace those in Exhibit D of this SOW, but supplement the guidance provided therein.)

- a. All furnace analyses shall fall within the calibration range. In addition, all analyses, except during full methods of standard addition (MSA), will require duplicate injections. The absorbance or concentration of each injection shall be reported in the raw data as well as the average absorbance or concentration values and the relative standard deviation (RSD) or coefficient of variation (CV). Average concentration values are used for reporting purposes. The Contractor shall be consistent per method and SDG in choosing absorbance or concentration to evaluate which route is to be followed in the MSA Tree. The Contractor shall also indicate which of the two is being used if both absorbance and concentration are reported in the raw data. For MSA analysis, the absorbance of each injection shall be included in the raw data. A maximum of 10 full sample analyses to a maximum 20 injections may be performed between each consecutive calibration verifications and blanks. For concentrations greater than CRDL, the duplicate injection readings must agree within 20% RSD or CV, or the analytical sample shall be rerun once (i.e., two additional burns). If the readings are still out, flag the value reported on FORM I-IN with an "M". The "M" flag is required for the analytical spike as well as the sample. If the analytical spike for a sample requires an "M" flag, the flag shall be reported on FORM I-IN for that sample.

FIGURE 1. FURNACE ATOMIC ABSORPTION ANALYSIS SCHEME



- b. All furnace analyses for each analytical sample, including those requiring an "M" flag, will require at least an analytical spike to determine if the MSA will be required for quantitation. The analytical spike⁶ will be required to be at a concentration (in the sample) 2x CRDL (except for lead which must be at 20 ug/L). This requirement for an analytical spike will include the LCS and the preparation blank. (The LCS shall be quantitated from the calibration curve and corrective action, if needed, taken accordingly. MSA is not to be performed on the LCS or preparation blank, regardless of spike recovery results.) If the preparation blank analytical spike recovery is out of control (85-115%), the spiking solution shall be verified by respiking and rerunning the preparation blank once. If the preparation blank analytical spike recovery is still out of control, correct the problem and reanalyze all analytical samples associated with that blank. An analytical spike shall not be performed on the matrix spike sample.

The analytical spike of a sample shall be run immediately after that sample. The percent recovery (%R) of the spike, calculated by the same formula as Spike Sample Analyses (see item 6, this section), will then determine how the sample will be quantitated, as follows:

- 1) If the spike recovery is less than 40%, the sample shall be diluted and rerun with another spike. Dilute the sample by a factor of 5 to 10 and rerun. This step shall only be performed once. If after the dilution the spike recovery is still <40%, report data and flag with an "E" to indicate interference problems.
- 2) If the spike recovery is greater than or equal to 40% and the sample absorbance or concentration is less than 50% of the "spike"⁷, report the sample results to the IDL. If the spike recovery is less than 85% or greater than 115%, flag the result with a "W".
- 3) If the sample absorbance or concentration is greater than or equal to 50% of the "spike"⁷ and the spike recovery is at or between 85% and 115%, the sample shall be quantitated directly from the calibration curve and reported down to the IDL.
- 4) If the sample absorbance or concentration is greater than or equal to 50% of the "spike"⁷ and the spike recovery is less

⁶Analytical spikes are furnace spikes to be prepared prior to analysis, but after digestion (if performed), by adding a known quantity of the analyte to an aliquot of the sample. The unspiked sample aliquot shall be compensated for any volume change in the spike samples by the addition of deionized water to the unspiked sample aliquot. The volume of the spiking solution added shall not exceed 10% of the analytical sample volume; this requirement also applies to MSA spikes.

⁷"Spike" is defined as [absorbance or concentration of spike sample] minus [absorbance or concentration of the sample].

than 85% or greater than 115%, the sample shall be quantitated by MSA.

c. The following procedures will be incorporated into MSA analyses.

- 1) Data from MSA calculations shall be within the linear range as determined by the calibration curve generated at the beginning of the analytical run.
- 2) The sample and three spikes shall be analyzed consecutively (MS0, MS1, MS2, MS3) for MSA quantitation (the "initial" spike run data are specifically excluded from use in the MSA quantitation). Only single injections shall be performed for MSA quantitation.

Each full MSA counts as two analytical samples towards determining 10% QC frequency (i.e., five full MSAs can be performed between calibration verifications).

- 3) For analytical runs containing only MSAs, single injections can be used for QC samples during that run. For instruments that operate in an MSA mode only, MSA can be used to determine QC samples during that run.
- 4) Spikes shall be prepared such that:
 - a) Spike 1 is approximately 50% of the sample concentration.
 - b) Spike 2 is approximately 100% of the sample concentration.
 - c) Spike 3 is approximately 150% of the sample concentration.
- 5) The data for each MSA analysis shall be clearly identified in the raw data documentation (using added concentration as the x-variable and absorbance as the y-variable) along with the slope, x-intercept, y-intercept and correlation coefficient (r) for the least squares fit of the data. The results shall be reported on FORM VIII-IN. Reported values obtained by MSA shall be flagged on the data sheet (FORM I-IN) with the letter "S" if the correlation coefficient is greater than or equal to 0.995.
- 6) If the correlation coefficient (r) for a particular analysis is less than 0.995, the MSA analysis shall be repeated once. If the correlation coefficient is still less than 0.995, report the results on FORM I-IN from the run with the best "r" and flag the result with a "+" on FORM VIII-IN and FORM I-IN.

SECTION VI

CONTRACT COMPLIANCE SCREENING

Contract Compliance Screening (CCS) is one aspect of the Government's contractual right of inspection of analytical data. CCS examines the Contractor's adherence to the contract requirements based on the sample data package delivered to the Agency.

CCS is performed by the Sample Management Office (SMO) under the direction of the EPA. To assure a uniform review, a set of standardized procedures has been developed to evaluate the sample data package submitted by a Contractor against the technical and completeness requirements of the contract.

CCS results are mailed to the Contractor and all other data recipients. The Contractor has a period of time to correct deficiencies. The Contractor shall send all corrections to the Regional Client and SMO/CLAS.

CCS results are used in conjunction with other information to measure overall Contractor performance and to take appropriate actions to correct deficiencies in performance.

The Agency may generate a CCS trend report which summarizes CCS results over a given period of time. The Agency may send the CCS trend report or discuss the CCS trend report during an on-site laboratory evaluation. In a detailed letter to the Technical Project Officer and Administrative Project Officer, the Contractor shall address the deficiencies and the subsequent corrective action implemented by the Contractor to correct the deficiencies within 14 days of receipt of the report or the on-site laboratory evaluation. An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of the Agency, represented by the Technical Project Officer or Administrative Project Officer, to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the Technical Project Officer, Administrative Project Officer, and Contracting Officer why he/she is unable to meet the delivery schedule listed in this section. The Technical Project Officer will not grant an extension for greater than 14 days for the Contractor's response to the CCS trend report. The Contractor shall proceed and not assume that an extension will be granted until so notified by the TPO and/or APO.

If new SOPs are required to be written, or if existing SOPs are required to be rewritten or amended because of deficiencies and subsequent corrective action implemented by the Contractor, the Contractor shall write/amend the SOPs per the requirements listed in Exhibit E, Section IV.

If the Contractor fails to adhere to the requirements listed in this section, the Contractor may expect, but the Agency is not limited to the following actions: reduction in the number of samples sent under the contract, suspension of sample shipment to the Contractor, data package audit, an on-site laboratory evaluation, a remedial performance evaluation sample, and/or contract sanctions, such as a Cure Notice.

SECTION VII

ANALYTICAL STANDARD REQUIREMENTS

The U.S. Environmental Protection Agency may be unable to supply analytical reference standards either for direct analytical measurements or for the purpose of traceability. In these cases, all contract laboratories will be required to prepare from materials or purchase from private chemical supply houses those standards necessary to successfully and accurately perform the analyses required in this protocol.

A. Preparation of Chemical Standards from the Neat High Purity Bulk Material

If the laboratory cannot obtain analytical reference data from the U.S. EPA, the laboratory may prepare their own chemical standards. Laboratories shall obtain the highest purity possible when purchasing chemical standards; standards purchased at less than 97% purity shall be documented as to why a higher purity could not be obtained.

1. If required by the manufacturer, the chemical standards shall be kept refrigerated when not being used in the preparation of standard solutions. Proper storage of chemicals is essential in order to safeguard them from decomposition.
2. The purity of a compound can sometimes be misrepresented by a chemical supply house. Since knowledge of purity is needed to calculate the concentration of solute in a solution standard, it is the contract laboratory's responsibility to have analytical documentation ascertaining that the purity of each compound is correctly stated. Purity confirmation, when performed, should use appropriate techniques. Use of two or more independent methods is recommended. The correction factor for impurity when weighing neat materials in the preparation of solution standards is:

Equation 1

$$\text{weight of impure compound} = \frac{\text{weight of pure compound}}{(\text{percent purity}/100)}$$

where "weight of pure compound" is that required to prepare a specific volume of a solution standard of a specified concentration.

3. Mis-identification of compounds occasionally occurs and it is possible that a mislabeled compound may be received from a chemical supply house. It is the contract laboratory's responsibility to have analytical documentation ascertaining that all compounds used in the preparation of solution standards are correctly identified.
4. Log notebooks are to be kept for all weighing and dilutions. All subsequent dilutions from the primary standard and the

calculations for determining their concentrations are to be recorded and verified by a second person. All solution standards are to be refrigerated, if required, when not in use. All solution standards are to be clearly labeled as to the identity of the analyte or analytes, concentration, date prepared, solvent, and initials of the preparer.

B. Purchase of chemical standards already in solution

1. Solutions of analytical reference standards can be purchased by Contractors provided they meet the following criteria:

Laboratories shall maintain documentation of the purity confirmation of the material to verify the integrity of the standard solutions they purchase.

2. The Contractor shall purchase standards for which the quality is demonstrated statistically and analytically by a method of the supplier's choice. One way this can be demonstrated is to prepare and analyze three solutions; a high standard, a low standard, and a standard at the target concentration (see parts a and b below). The supplier must then demonstrate that the analytical results for the high standard and low standard are consistent with the difference in theoretical concentrations. This is done by the Student's t-test in part "d". If this is achieved, the supplier must then demonstrate that the concentration of the target standard lies midway between the concentrations of the low and high standards. This is done by the Student's t-test in part e. Thus the standard is certified to be within 10 percent of the target concentration.

If the procedure above is used, the supplier must document that the following have been achieved:

- a. Two solutions of identical concentration shall be prepared independently from neat materials. An aliquot of the first solution shall be diluted to the intended concentration (the "target standard"). One aliquot is taken from the second solution and diluted to a concentration ten percent greater than the target standard. This is called the "high standard". One further aliquot is taken from the second solution and diluted to a concentration 10 percent less than the target standard. This is called the "low standard".
- b. Six replicate analyses of each standard (a total of 18 analyses) shall be performed in the following sequence: low standard, target standard, high standard, low standard, target standard, high standard, ...

- c. The mean and variance of the six results for each solution shall be calculated.

Equation 2

$$MEAN = \frac{Y_1 + Y_2 + Y_3 + Y_4 + Y_5 + Y_6}{6}$$

Equation 3

$$VARIANCE = \frac{Y_1^2 + Y_2^2 + Y_3^2 + Y_4^2 + Y_5^2 + Y_6^2 - (6 * MEAN)^2}{5}$$

The values Y_1, Y_2, Y_3, \dots , represent the results of the six analyses of each standard. The means of the low, target, and high standards are designated M_1, M_2 , and M_3 , respectively. The variances of the low, target, and high standards are designated V_1, V_2 , and V_3 , respectively. Additionally, a pooled variance, V_p , is calculated.

Equation 4

$$V_p = \frac{\frac{V_1}{0.81} + V_2 + \frac{V_3}{1.21}}{3}$$

If the square root of V_p is less than one percent of M_2 , then $M_2^2 / 10,000$ is to be used as the value of V_p in all subsequent calculations.

- d. The test statistic shall be calculated:

Equation 5

$$TEST\ STATISTIC = \frac{\left| \frac{M_3}{1.1} - \frac{M_1}{0.9} \right|}{\left(\frac{V_p}{3} \right)^{0.5}}$$

If the test statistic exceeds 2.13, then the supplier has failed to demonstrate a twenty percent difference between the high and low standards. In such a case, the standards are not acceptable.

- e. The test statistic shall be calculated:

Equation 6

$$TEST\ STATISTIC = \frac{\left| M_2 - \left(\frac{M_1}{1.8} \right) - \left(\frac{M_3}{2.2} \right) \right|}{\left(\frac{V_P}{4} \right)^{0.5}}$$

If the test statistic exceeds 2.13, the supplier has failed to demonstrate that the target standard concentration is midway between the high and low standards. In such a case, the standards are not acceptable.

- f. The 95 percent confidence intervals for the mean result of each standard shall be calculated:

Equation 7

$$Interval\ for\ Low\ Standard = M_1 \pm 2.13 \left(\frac{V_P}{6} \right)^{0.5}$$

Equation 8

$$Interval\ for\ Target\ Standard = M_2 \pm 2.13 \left(\frac{V_P}{6} \right)^{0.5}$$

Equation 9

$$Interval\ for\ High\ Standard = M_3 \pm 2.13 \left(\frac{V_P}{6} \right)^{0.5}$$

These intervals shall not overlap. If overlap is observed, then the supplier has failed to demonstrate the ability to discriminate the 10 percent difference in concentrations. In such a case, the standards are not acceptable. In any event, the laboratory is responsible for the quality of the standards employed for analyses under this contract.

C. Requesting Standards From the EPA Standards Repository

Solutions of analytical reference materials can be ordered from the U.S. EPA Chemical Standards Repository, depending on availability. The Contractor can place an order for standards only after demonstrating that these standards are not available from commercial vendors either in solution or as a neat material.

D. Documentation of the Verification and Preparation of Chemical Standards

It is the responsibility of each laboratory to maintain the necessary documentation to show that the chemical standards they have used in the performance of CLP analysis conform to the requirements previously listed. Weighing logbooks, calculations, raw data, etc., whether produced by the laboratory or purchased from chemical supply houses, shall be maintained by the laboratory and may be subject to review during on-site inspection visits. In those cases where the documentation is supportive of the analytical results of data packages sent to EPA, such documentation is to be kept on file by the laboratories for a period of one year.

Upon request by the Technical Project Officer or Administrative Project Officer, the Contractor shall submit their most recent previous year's documentation (12 months) for the verification and preparation of chemical standards within 14 days of the receipt of request to the recipients he/she designates.

The Agency may generate a report discussing deficiencies in the Contractor's documentation for the verification and preparation of chemical standards or may discuss the deficiencies during an on-site laboratory evaluation. In a detailed letter to the Technical Project Officer, Administrative Project Officer, and EMSL/LV, the Contractor shall address the deficiencies and the subsequent corrective action implemented by the Contractor to correct the deficiencies within 14 days of receipt of the report or the on-site laboratory evaluation. An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of the Agency, represented either by the Technical Project Officer or Administrative Project Officer, to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the Technical Project Officer, Administrative Project Officer, and the Contracting Officer why he/she is unable to meet the delivery schedule listed in this section. The Technical Project Officer/Administrative Project Officer will not grant an extension for greater than 14 days for the Contractor's response letter to the standards documentation report. The Contractor shall proceed and not assume that an extension will be granted until so notified by the TPO and/or APO.

If new SOPs are required to be written, or if existing SOPs are required to be rewritten or amended because of deficiencies and subsequent corrective action implemented by the Contractor, the Contractor shall write/amend the SOPs per the requirements listed in Exhibit E, Section IV.

If the Contractor fails to adhere to the requirements listed in this Section, a Contractor may expect, but the Agency is not limited to the following actions: reduction in the number of samples sent under the contract, suspension of sample shipment to Contractor, data package audit, an on-site laboratory evaluation, a remedial laboratory evaluation sample, and/or contract sanctions, such as a Cure Notice.

SECTION VIII

DATA PACKAGE AUDITS

Data package audits are performed by the Agency for program overview and specific Regional concerns. Standardized procedures have been established to assure uniformity of the auditing process. Data packages are periodically selected from recently received Cases. They are evaluated for the technical quality of hardcopy raw data, quality assurance, and the adherence to contractual requirements. This function provides external monitoring of program QC requirements.

Data package audits are used to assess the technical quality of the data and evaluate overall laboratory performance. Audits provide the Agency with an in-depth inspection and evaluation of the Case data package with regard to achieving QA/QC acceptability. A thorough review of the raw data is completed including: all instrument readouts used for the sample results, instrument printouts, and other documentation for deviations from the contractual requirements, a check for transcription and calculation errors, a review of the qualifications of the laboratory personnel involved with the Case, and a review of all current SOPs on file.

Responding to the Data Package Audit Report:

After completion of the data package audit, the Agency may send a copy of the data package audit report to the Contractor or may discuss the data package audit report on an on-site laboratory evaluation. In a detailed letter to the Technical Project Officer, Administrative Project Officer, and the EPA designated recipient, the Contractor shall discuss the corrective actions implemented to resolve the deficiencies listed in the data package audit report within 14 days of receipt of the report. An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of the Agency, represented either by the Technical Project Officer or Administrative Project Officer, to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the Technical Project Officer, Administrative Project Officer, and the Contracting Officer why he/she is unable to meet the delivery schedule listed in this section. The Technical Project Officer/Administrative Project Officer will not grant an extension for greater than 14 days for the Contractor's response letter to the data package report. The Contractor shall proceed and not assume that an extension will be granted until so notified by the TPO and/or APO.

If new SOPs are required to be written, or if existing SOPs are required to be rewritten or amended because of deficiencies and subsequent corrective action implemented by the Contractor, the Contractor shall write/amend the SOPs per the requirements listed in Exhibit E, Section IV.

Corrective Action:

If the Contractor fails to adhere to the requirements listed in this section, the Contractor may expect, but the Agency is not limited to the following actions: reduction in the number of samples sent under the contract,

suspension of sample shipment to the Contractor, an on-site laboratory evaluation, data package audit, remedial performance evaluation sample, and/or contract sanctions, such as a Cure Notice.

Regional Data Review:

Contractor data are generated to meet the specific needs of the EPA Regions. In order to verify the useability of data for the intended purpose, each Region reviews data from the perspective of the end user, based on functional guidelines for data review which have been developed jointly by the Regions and the National Program Office. Each Region uses these guidelines as the basis for data evaluation. Individual Regions may augment the basic guideline review process with additional review based on Region-specific or site-specific concerns. Regional reviews, like the sites under investigation, vary based on the nature of the problem under investigation and the Regional response appropriate to the specific circumstances.

Regional data reviews, relating usability of the data to a specific site, are part of the collective assessment process. They complement the review done at the Sample Management Office, which is designed to identify contractual discrepancies. These individual evaluations are integrated into collective review that is necessary for Program and Contractor administration and management and may be used to take appropriate action to correct deficiencies in the Contractor's performance.

SECTION IX

PERFORMANCE EVALUATION SAMPLES

Although intralaboratory QC may demonstrate Contractor and method performance that can be tracked over time, an external performance evaluation program is an essential feature of a QA program. As a means of measuring Contractor and method performance, Contractors participate in interlaboratory comparison studies conducted by the EPA. Results from the analysis of these performance evaluation samples (PES) will be used by the EPA to verify the Contractor's continuing ability to produce acceptable analytical data. The results are also used to assess the precision and accuracy of the analytical methods for specific analytes.

Sample sets may be provided to participating Contractors as frequently as on an SDG-by-SDG basis as a recognizable QC sample of known composition; as a recognizable QC sample of unknown composition; or not recognizable as a QC material. The laboratory evaluation samples may be sent either by the Regional client or the National Program Office. The results of all such quality control samples may be used as the basis for rejection of data for: sample(s) within an SDG, a fraction (e.g., metals and/or cyanide) within an SDG or the entire SDG, and/or may be used as the basis for contract action. The Contractor shall analyze the samples and return the data package and all raw data within the contract required turnaround time.

In addition to PES preparation and analysis, the Contractor will be responsible for correctly identifying and quantifying the analytes included in the PES. The Agency will notify the Contractor of unacceptable performance.

Contractors are required to analyze the samples and return the data package and all raw data within the contract required turnaround time.

A Contractor's results on the laboratory evaluation samples will determine the Contractor's performance as follows:

1. Acceptable, No Response Required (Score greater than or equal to 90 percent):

Data meets most or all of the scoring criteria. No response is required.

2. Acceptable, Response Explaining Deficiency(ies) Required (Score greater than or equal to 75 percent but less than 90 percent):

Deficiencies exist in the Contractor's performance.

Within 14 days of receipt of notification from EPA, the Contractor shall describe the deficiency(ies) and the action(s) taken to correct the deficiency(ies) in a letter to the Administrative Project Officer, the Technical Project Officer and the EPA designated recipient.

An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of the Agency, represented either by the Technical Project Officer or Administrative Project Officer, to approve or

disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the Technical Project Officer, Administrative Project Officer, and the Contracting Officer why he/she is unable to meet the delivery schedule listed in this section. The Technical Project Officer/Administrative Project Officer will not grant an extension for greater than 14 days for the Contractor's response letter to the laboratory evaluation sample report. The Contractor shall proceed and not assume that an extension will be granted until so notified by the TPO and/or APO.

If new SOPs are required to be written or if existing SOPs are required to be rewritten or amended because of deficiencies and subsequent corrective action implemented by the Contractor, the Contractor shall write/amend the SOPs per the requirements listed in Exhibit E, Section IV.

3. Unacceptable Performance, Response Explaining Deficiency(ies) Required
(Score less than 75 percent):

Deficiencies exist in the Contractor's performance to the extent that the National Program Office has determined that the Contractor has not demonstrated the capability to meet the contract requirements.

Within 14 days of receipt of notification from EPA, the Contractor shall describe the deficiency(ies) and the action(s) taken to correct the deficiency(ies) in a letter to the Administrative Project Officer, the Technical Project Officer and the EPA designated recipient.

An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of the Agency, represented either by the Technical Project Officer or Administrative Project Officer, to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the Technical Project Officer, Administrative Project Officer, and the Contracting Officer why he/she is unable to meet the delivery schedule listed in this section. The Technical Project Officer/Administrative Project Officer will not grant an extension for greater than 14 days for the Contractor's response letter to the performance evaluation sample report. The Contractor shall proceed and not assume that an extension will be granted until so notified by the TPO and/or APO.

If new SOPs are required to be written, or if existing SOPs are required to be rewritten or amended because of deficiencies and subsequent corrective action implemented by the Contractor, the Contractor shall write/amend the SOPs per the requirements listed in Exhibit E, Section IV.

The Contractor shall be notified by the Technical Project Officer or Administrative Project Officer concerning the remedy for their unacceptable performance. A Contractor may expect, but the Agency is not limited to, the following actions: reduction in the number of samples sent under the contract, suspension of sample shipment to the Contractor, an on-site laboratory evaluation, data package audit, remedial performance evaluation sample, and/or a contract sanction, such as a Cure Notice.

Note: A Contractor's prompt response demonstrating that corrective actions have been taken to ensure the Contractor's capability to meet contract requirements may facilitate continuation of full sample delivery.

If the Contractor fails to adhere to the requirements listed in this section, a Contractor may expect, but the Agency is not limited to the following actions: reduction in the number of samples sent under the contract, suspension of sample shipment to the Contractor, an on-site laboratory evaluation, data package audit, a remedial laboratory evaluation sample and/or contract sanctions, such as a Cure Notice.

SECTION X

ON-SITE LABORATORY EVALUATIONS

At a frequency dictated by a contract laboratory's performance, the Administrative Project Officer, Technical Project Officer or their authorized representative will conduct an on-site laboratory evaluation. On-site laboratory evaluations are carried out to monitor the Contractor's ability to meet selected terms and conditions specified in the contract. The evaluation process incorporates two separate categories: Quality Assurance Evaluation and an Evidentiary Audit.

A. Quality Assurance On-Site Evaluation

Quality assurance evaluators inspect the Contractor's facilities to verify the adequacy and maintenance of instrumentation, the continuity, experience and education of personnel, and the acceptable performance of analytical and QC procedures. The Contractor should expect that items to be monitored will include, but not be limited to, the following:

- Size and appearance of the facility
- Quantity, age, availability, scheduled maintenance and performance of instrumentation
- Availability, appropriateness, and utilization of the QAP and SOPs
- Staff qualifications, experience, and personnel training programs
- Reagents, standards, and sample storage facilities
- Standard preparation logbooks and raw data
- Bench sheets and analytical logbook maintenance and review
- Review of the Contractor's sample analysis/data package inspection/data management procedures

Prior to an on-site evaluation, various documentation pertaining to performance of the specific Contractor is integrated in a profile package for discussion during the evaluation. Items that may be included are previous on-site reports, performance evaluation sample scores, Regional review of data, Regional QA materials, data audit reports, results of CCS, and data trend reports.

B. Evidentiary Audit

Evidence auditors conduct an on-site laboratory evaluation to determine if laboratory policies and procedures are in place to satisfy evidence handling requirements as stated in Exhibit F. The evidence audit is comprised of the following three activities:

1. Procedural Audit

The procedural audit consists of review and examination of actual standard operating procedures and accompanying documentation for the following laboratory operations: sample receiving, sample storage, sample identification, sample security, sample tracking (from receipt to completion of analysis) and analytical project file organization and assembly.

2. Written SOPs Audit

The written SOPs audit consists of review and examination of the written SOPs to determine if they are accurate and complete for the following laboratory operations: sample receiving, sample storage, sample identification, sample security, sample tracking (from receipt to completion of analysis) and analytical project file organization and assembly.

3. Analytical Project File Evidence Audit

The analytical project file evidence audit consists of review and examination of the analytical project file documentation. The auditors review the files to determine:

- The accuracy of the document inventory
- The completeness of the file
- The adequacy and accuracy of the document numbering system
- Traceability of sample activity
- Identification of activity recorded on the documents
- Error correction methods

C. Discussion of the On-Site Team's Findings

The quality assurance and evidentiary auditors discuss their findings with the Administrative Project Officer/Technical Project Officer prior to debriefing the Contractor. During the debriefing, the auditors present their findings and recommendations for corrective actions necessary to the Contractor personnel.

D. Corrective Action Reports For Follow-Through to Quality Assurance and Evidentiary Audit Reports

On-site laboratory evaluation:

Following an on-site laboratory evaluation, quality assurance and/or evidentiary audit reports which discuss deficiencies found during the on-site evaluation may be sent to the Contractor. In a detailed letter, the Contractor shall discuss the corrective actions implemented to resolve the deficiencies discussed during the on-site evaluation and

discussed in the report(s) to the Technical Project Officer and Administrative Project Officer within 14 days of receipt of the report. An alternate delivery schedule may be proposed by the Contractor, but it is the sole decision of the Agency, represented either by the Technical Project Officer or Administrative Project Officer, to approve or disapprove the alternate delivery schedule. If an alternate delivery schedule is proposed, the Contractor shall describe in a letter to the Technical Project Officer, Administrative Project Officer, and the Contracting Officer why he/she is unable to meet the delivery schedule listed in this section. The Technical Project Officer/Administrative Project Officer will not grant an extension for greater than 14 days for the Contractor's response letter to the quality assurance and evidentiary audit report. The Contractor shall proceed and not assume that an extension will be granted until so notified by the TPO and/or APO.

If new SOPs are required to be written, or if existing SOPs are required to be rewritten or amended because of the deficiencies and the subsequent corrective action implemented by the Contractor, the Contractor shall write/amend the SOPs per the requirements listed in Exhibit E, Section IV.

Corrective Action:

If the Contractor fails to adhere to the requirements listed in this section, the Contractor may expect, but the Agency is not limited to the following actions: reduction in the number of samples sent under the contract, suspension of sample shipment to the Contractor, an on-site laboratory evaluation, data package audit, a remedial performance evaluation sample, and/or contract sanctions, such as a Cure Notice.

SECTION XI

DATA MANAGEMENT

Data management procedures are defined as procedures specifying the acquisition or entry, update, correction, deletion, storage and security of computer readable data and files. These procedures shall be in written form and contain a clear definition for all databases and files used to generate or resubmit deliverables. Key areas of concern include: system organization (including personnel and security), documentation operations, traceability and quality control.

Data manually entered from hard-copy shall be quality controlled and the error rates estimated. Systems should prevent entry of incorrect or out-of-range data and alert data entry personnel of errors. In addition, data entry error rates shall be estimated and recorded on a monthly basis by reentering a statistical sample of the data entered and calculating discrepancy rates by data element.

The record of changes in the form of corrections and updates to data originally generated, submitted, and/or resubmitted shall be documented to allow traceability of updates. Documentation shall include the following for each change:

- Justification or rationale for the change.
- Initials of the person making the change or changes. Data changes shall be implemented and reviewed by a person or group independent of the source generating the deliverable.
- Change documentation shall be retained according to the schedule of the original deliverable.
- Resubmitted diskettes or other deliverables shall be reinspected as a part of the laboratory's internal inspection process prior to resubmission. The entire deliverable, not just the changes, shall be inspected.
- The Laboratory Manager shall approve changes to originally submitted deliverables.
- Documentation of data changes may be requested by laboratory auditors.

Lifecycle management procedures shall be applied to computer software systems developed by the laboratory to be used to generate and edit contract deliverables. Such systems shall be thoroughly tested and documented prior to utilization.

- A software test and acceptance plan including test requirements, test results and acceptance criteria shall be developed, followed, and available in written form.

- System changes shall not be made directly to production systems generating deliverables. Changes shall be made first to a development system and tested prior to implementation.
- Each version of the production system will be given an identification number, date of installation, and date of last operation and will be archived.
- System and operations documentation shall be developed and maintained for each system. Documentation shall include a user's manual and an operations and maintenance manual.

Individual(s) responsible for the following functions shall be identified:

- System operation and maintenance including documentation and training.
- Database integrity, including data entry, data updating and quality control.
- Data and system security, backup and archiving.

EXHIBIT F

CHAIN-OF-CUSTODY, DOCUMENT CONTROL,
AND WRITTEN STANDARD OPERATING PROCEDURES

1. INTRODUCTION

1.1 A sample is physical evidence collected from a facility or from the environment. Controlling evidence is an essential part of the hazardous waste investigation effort. To ensure that EPA's sample data and records supporting sample-related activities are admissible and have weight as evidence in future litigation, Contractors are required to maintain EPA samples under chain-of-custody and to account for all samples and supporting records of sample handling, preparation, and analysis. Contractors shall maintain sample identity, sample custody, and all sample-related records according to the requirements in this exhibit.

1.2 The purposes of the evidence requirements include:

- Ensuring traceability of samples while in possession of the Contractor.
- Ensuring custody of samples while in possession of the Contractor.
- Ensuring the integrity of sample identity while in possession of the Contractor.
- Ensuring sample-related activities are recorded on documents or in other formats for EPA sample receipt, storage, preparation, analysis, and disposal.
- Ensuring all laboratory records for each specified Sample Delivery Group will be accounted for when the project is completed.
- Ensuring that all laboratory records directly related to EPA samples are assembled and delivered to EPA or, prior to delivery, are available upon EPA's request.

2. Standard Operating Procedures

The Contractor shall implement the following standard operating procedures for sample receiving, sample identification, sample security, sample storage, sample tracking and document control, computer-resident sample data control, and Complete Sample Delivery Group File organization and assembly to ensure accountability of EPA sample chain-of-custody as well as control of all EPA sample-related records.

2.1 Sample Receiving

2.1.1 The Contractor shall designate a sample custodian responsible for receiving EPA samples.

2.1.2 The Contractor shall designate a representative to receive EPA samples in the event that the sample custodian is not available.

- 2.1.3 Upon receipt, the condition of shipping containers and sample containers shall be inspected and recorded on Form DC-1 by the sample custodian or his/her representative.
- 2.1.4 Upon receipt, the condition of the custody seals (intact/broken) shall be inspected and recorded on Form DC-1 by the sample custodian or his/her representative.
- 2.1.5 The sample custodian or his/her representative shall verify and record on Form DC-1 the presence or absence of the following documents accompanying the sample shipment:
- Custody seals,
 - Chain-of-custody records,
 - Traffic reports or packing lists,
 - Airbills or airbill stickers, and
 - Sample tags.
- 2.1.6 The sample custodian or his/her representative shall verify and record on Form DC-1 the agreement or disagreement of information recorded on all documents received with samples and information recorded on sample containers.
- 2.1.7 The sample custodian or his/her representative shall record the following information on Form DC-1 as samples are received and inspected:
- Custody seal numbers when present,
 - Airbill or airbill sticker numbers,
 - Sample tags listed/not listed on chain-of-custody records,
 - Date of receipt,
 - Time of receipt,
 - EPA sample numbers,
 - Sample tag numbers,
 - Assigned laboratory numbers,
 - Samples delivered by hand, and
 - Problems and discrepancies.
- 2.1.8 The sample custodian or his/her representative shall sign, date, and record the time on all accompanying forms, when applicable, at the time of sample receipt (for example, chain-of-custody records, traffic reports or packing lists, and airbills).

Note: Initials are not acceptable.

2.1.9 The Contractor shall contact the Sample Management Office (SMO) to resolve problems and discrepancies including, but not limited to, absent documents, conflicting information, absent or broken custody seals, and unsatisfactory sample condition (for example, leaking sample container).

2.1.10 The Contractor shall record resolution of problems and discrepancies by SMO.

2.2 Sample Identification

2.2.1 The Contractor shall maintain the identity of EPA samples and prepared samples (including extracted samples, digested samples, and distilled samples) throughout the laboratory.

2.2.2 Each sample and sample preparation container shall be labeled with the EPA number or a unique laboratory sample identification number.

2.3 Sample Security

2.3.1 The Contractor shall demonstrate that EPA sample custody is maintained from receiving through retention or disposal. A sample is in custody if:

- It is in your possession, or
- It is in your view after being in your possession, or
- It is locked in a secure area after being in your possession, or
- It is in a designated secure area. (Secure areas shall be accessible only to authorized personnel.)

2.3.2 The Contractor shall demonstrate security of designated secure areas.

2.4 Sample Storage

The Contractor shall designate storage areas for EPA samples and prepared samples.

2.5 Sample Tracking and Document Control

2.5.1 The Contractor shall record all activities performed on EPA samples.

2.5.2 Titles which identify the activities recorded shall be printed on each page of all laboratory documents. (Activities include, but are not limited to, sample receipt, sample storage, sample preparation, and sample analysis.) When a document is a record

of analysis, the instrument type and parameter group (for example, ICP-Metals) shall be included in the title.

- 2.5.3 When columns are used to organize information recorded on laboratory documents, the information recorded in the columns shall be identified in a column heading.
- 2.5.4 Reviewers' signatures shall be identified on laboratory documents when reviews are conducted.

Note: Individuals recording review comments on computer-generated raw data are not required to be identified unless the written comments address data validity.

- 2.5.5 The laboratory name shall be identified on preprinted laboratory documents.
- 2.5.6 Each laboratory document entry shall be dated with the month/day/year (for example, 01/01/90) and signed (or initialed) by the individual(s) responsible for performing the recorded activity at the time the activity is recorded.
- 2.5.7 Notations on laboratory documents shall be recorded in ink.
- 2.5.8 Corrections to laboratory documents and raw data shall be made by drawing single lines through the errors and entering the correct information. Information shall not be obliterated or rendered unreadable. Corrections and additions to information shall be signed (or initialed) and dated.
- 2.5.9 Unused portions of laboratory documents shall be lined-out.
- 2.5.10 Pages in bound and unbound logbooks shall be sequentially numbered.
- 2.5.11 Instrument-specific run logs shall be maintained to enable the reconstruction of run sequences.
- 2.5.12 Logbook entries shall be in chronological order.
- 2.5.13 Logbook entries shall include only one Sample Delivery Group (SDG) per page, except in the events where SDGs "share" QC samples (for example, instrument run logs and extraction logs).
- 2.5.14 Information inserted into laboratory documents shall be affixed permanently in place. The individual responsible for inserting information shall sign and date across the insert and logbook page at the time information is inserted.
- 2.5.15 The Contractor shall document disposal or retention of EPA samples, remaining portions of samples, and prepared samples.

2.6 Computer-Resident Sample Data Control

- 2.6.1 Contractor personnel responsible for original data entry shall be identified at the time of data input.
- 2.6.2 The Contractor shall make changes to electronic data in a manner which ensures that the original data entry is preserved, the editor is identified, and the revision date is recorded.
- 2.6.3 The Contractor shall routinely verify the accuracy of manually entered data, electronically entered data, and data acquired from instruments.
- 2.6.4 The Contractor shall routinely verify documents produced by the electronic data collection system to ensure accuracy of the information reported.
- 2.6.5 The Contractor shall ensure that the electronic data collection system is secure.
 - The electronic data collection system shall be maintained in a secure location.
 - Access to the electronic data collection system functions shall be limited to authorized personnel through utilization of software security techniques (for example, log-ons or restricted passwords).
 - Electronic data collection systems shall be protected from the introduction of external programs or software (for example, viruses).
- 2.6.6 The Contractor shall designate archive storage areas for electronic data and the software required to access the data.
- 2.6.7 The Contractor shall designate an individual responsible for maintaining archives of electronic data including the software.
- 2.6.8 The Contractor shall maintain the archives of electronic data and necessary software in a secure location. (Secure areas shall be accessible only to authorized personnel.)

2.7 Complete Sample Delivery Group File Organization and Assembly

- 2.7.1 The Contractor shall designate a document control officer responsible for the organization and assembly of the Complete SDG File (CSF).
- 2.7.2 The Contractor shall designate a representative responsible for the organization and assembly of the CSF in the event that the document control officer is not available.
- 2.7.3 The Contractor shall maintain documents relating to the CSF in a secure location.

- 2.7.4 All original laboratory forms and copies of SDG-related logbook pages shall be included in the CSF.
- 2.7.5 Copies of laboratory documents in the CSF shall be photocopied in a manner to provide complete and legible replicates.
- 2.7.6 Documents relevant to each SDG including, but not limited to, the following shall be included in the CSF:
- logbook pages,
 - benchsheets,
 - screening records,
 - preparation records,
 - re-preparation records,
 - analytical records,
 - re-analysis records,
 - records of failed or attempted analysis,
 - custody records,
 - sample tracking records,
 - raw data summaries,
 - computer printouts,
 - correspondence,
 - FAX originals,
 - library search results, and
 - other.
- 2.7.7 The document control officer or his/her representative shall ensure that sample tags are encased in clear plastic bags before placing them in the CSF.
- 2.7.8 CSF documents shall be organized and assembled on an SDG-specific basis.
- 2.7.9 Original documents which include information relating to more than one SDG (for example, chain-of-custody records, traffic reports, calibration logs) shall be filed in the CSF of the lowest SDG number, and copies of these originals shall be placed in the other CSF(s). The document control officer or his/her representative shall record the following statement on the copies in dark ink:

COPY
ORIGINAL DOCUMENTS ARE INCLUDED IN CSF _____

Signature

Date

- 2.7.10 All CSFs shall be submitted with a completed Form DC-2. All resubmitted CSFs shall be submitted with a new or revised Form DC-2.
- 2.7.11 Each item in the CSF and resubmitted CSFs shall be inventoried and assembled in the order specified on Form DC-2. Each page of the CSF shall be stamped with a sequential number. Page number ranges shall be recorded in the columns provided on Form DC-2.

Intentional gaps in the page numbering sequence shall be recorded in the "Comments" section on Form DC-2. When inserting new or inadvertently omitted documents, the Contractor shall identify them with unique accountable numbers. The unique accountable numbers and the locations of the documents shall be recorded in the "Other Records" section on Form DC-2.

2.7.12 Before shipping each CSF, the document control officer or his/her representative shall verify the agreement of information recorded on all documentation and ensure that the information is consistent and the CSF is complete.

2.7.13 The document control officer or his/her representative shall document the shipment of deliverable packages including what was sent, to whom, the date, and the carrier used.

2.7.14 Shipments of deliverable packages, including resubmittals, shall be sealed with custody seals by the document control officer or his/her representative in a manner such that opening the packages would break the seals.

2.7.15 Custody seals shall be signed and dated by the document control officer or his/her representative when sealing deliverable packages.

3. WRITTEN STANDARD OPERATING PROCEDURES

The Contractor shall develop and implement the following written standard operating procedures (SOPs) for sample receiving, sample identification, sample security, sample storage, sample tracking and document control, computer-resident sample data control, and CSF file organization and assembly to ensure accountability for EPA sample chain-of-custody and control of all EPA sample-related records.

3.1 Sample Receiving

3.1.1 The Contractor shall have written SOPs for sample receiving which accurately reflect the procedures used by the laboratory.

3.1.2 The written SOPs for sample receiving shall ensure that the procedures listed below are in use at the laboratory.

- The condition of shipping containers and sample containers are inspected and recorded on Form DC-1 upon receipt by the sample custodian or his/her representative.
- The condition of custody seals are inspected and recorded on Form DC-1 upon receipt by the sample custodian or his/her representative.
- The presence or absence of the following documents accompanying the sample shipment is verified and recorded on Form DC-1 by the sample custodian or his/her representative:

-- Custody seals,

- Chain-of-custody records,
- Traffic reports or packing lists,
- Airbills or airbill stickers, and
- Sample tags.
- The agreement or disagreement of information recorded on shipping documents with information recorded on sample containers is verified and recorded on Form DC-1 by the sample custodian or his/her representative.
- The following information is recorded on Form DC-1 by the sample custodian or his/her representative as samples are received and inspected:
 - Custody seal numbers when present,
 - Airbill or airbill sticker numbers,
 - Sample tag numbers listed/not listed on chain-of-custody records,
 - Date of receipt,
 - Time of receipt,
 - EPA sample numbers,
 - Sample tag numbers,
 - Assigned laboratory numbers,
 - Samples delivered by hand, and
 - Problems and discrepancies.
- All accompanying forms are signed, dated, and the time is recorded, when applicable, at the time of sample receipt (for example, chain-of-custody records, traffic reports or packing lists, and airbills) by the sample custodian or his/her representative.
- SMO is contacted to resolve problems and discrepancies including, but not limited to, absent documents, conflicting information, absent or broken custody seals, and unsatisfactory sample condition (for example, leaking sample container).
- The resolution of problems and discrepancies by SMO is recorded.

3.2 Sample Identification

3.2.1 The Contractor shall have written SOPs for sample identification which accurately reflect the procedures used by the laboratory.

3.2.2 The written SOPs for sample identification shall ensure that the procedures listed below are in use at the laboratory.

- The identity of EPA samples and prepared samples is maintained throughout the laboratory:
 - When the Contractor assigns unique laboratory sample identification numbers, the written SOPs shall include a description of the procedure used to assign these numbers,
 - When the Contractor uses prefixes or suffixes in addition to laboratory sample identification numbers, the written SOPs shall include their definitions, and
 - When the Contractor uses methods to uniquely identify fractions/parameter groups and matrix type, the written SOPs shall include a description of these methods.
- Each sample and sample preparation container is labeled with the SMO number or a unique laboratory sample identification number.

3.3 Sample Security

3.3.1 The Contractor shall have written SOPs for sample security which accurately reflect the procedures used by the laboratory.

3.3.2 The written SOPs for sample security shall include the items listed below.

- Procedures which ensure the following:
 - Sample custody is maintained, and
 - The security of designated secure areas is maintained.
- A list of authorized personnel who have access to locked storage areas.

3.4 Sample Storage

3.4.1 The Contractor shall have written SOPs for sample storage which accurately reflect the procedures used by the laboratory.

3.4.2 The written SOPs for sample storage shall describe locations, contents, and identities of all storage areas for EPA samples and prepared samples in the laboratory.

3.5 Sample Tracking and Document Control

3.5.1 The Contractor shall have written SOPs for sample tracking and document control which accurately reflect the procedures used by the laboratory.

3.5.2 The written SOPs for sample tracking and document control shall include the items listed below.

- Examples of all laboratory documents used during sample receiving, sample storage, sample transfer, sample analyses, CSF organization and assembly, and sample retention or disposal.
- Procedures which ensure the following:
 - All activities performed on EPA samples are recorded;
 - Titles which identify the activities recorded are printed on each page of all laboratory documents;
 - Information recorded in columns is identified with column headings;
 - Reviewers' signatures are identified on laboratory documents;
 - The laboratory name is included on preprinted laboratory documents;
 - Laboratory document entries are signed and dated with the month/day/year (for example, 01/01/90);
 - Entries on all laboratory documents are recorded in ink;
 - Corrections and additions to laboratory documents are made by drawing single lines through the errors, entering the correct information, and initialing and dating the new information;
 - Unused portions of laboratory documents are lined-out;
 - Pages in bound and unbound logbooks are sequentially numbered;
 - Instrument-specific run logs are maintained to enable the reconstruction of run sequences;
 - Logbook entries are recorded in chronological order;
 - Entries are recorded for only one SDG on a page, except in the event where SDGs "share" QC samples (for example, instrument run logs and extraction logs);

- Information inserted in laboratory documents is affixed permanently, signed or initialled, and dated across the insert; and
- The retention or disposal of EPA samples, remaining portions of samples, and prepared samples is documented.

3.6 Computer-Resident Sample Data Control

3.6.1 The Contractor shall have written SOPs for computer-resident sample data control which accurately reflect the procedures used by the laboratory.

3.6.2 The written SOPs for computer-resident sample data control shall include the items listed below.

- Procedures which ensure the following:
 - Contractor personnel responsible for original data entry are identified;
 - Changes to electronic data are made such that the original data entry is preserved, the editor is identified, and the revision date is recorded;
 - The accuracy of manually entered data, electronically entered data, and data acquired from instruments is verified;
 - Report documents produced by the electronic data collection system are routinely verified to ensure the accuracy of the information reported;
 - Electronic data collection system security is maintained; and
 - Archives of electronic data and accompanying software are maintained in a secure location.
- Descriptions of archive storage areas for the electronic data and the software required to access data archives.
- A list of authorized personnel who have access to electronic data collection system functions and to archived data.

3.7 CSF Organization and Assembly

3.7.1 The Contractor shall have written SOPs for CSF organization and assembly which accurately reflect the procedures used by the laboratory.

3.7.2 The written SOPs for CSF organization and assembly shall ensure that the procedures listed below are in use at the laboratory.

- Documents relating to the CSF are maintained in a secure location.
- All original laboratory forms and copies of SDG-related logbook pages are included in the CSF.
- Laboratory documents are photocopied in a manner to provide complete and legible replicates.
- All documents relevant to each SDG are included in the CSF.
- Sample tags are encased in clear plastic bags by the document control officer or his/her representative before placing them in the CSF.
- The CSF is organized and assembled on an SDG-specific basis.
- Original documents which contain information relating to more than one SDG are filed in the CSF of the lowest SDG and copies are referenced to originals in the event that an original document contains information relating to more than one SDG.
- Each CSF is submitted with a completed Form DC-2, and resubmitted CSFs are submitted with a new or revised Form DC-2.
- Each page of the CSF is stamped with a sequential number and the page number ranges are recorded in the columns provided on Form DC-2. Intentional gaps in the page numbering sequence are recorded in the "Comments Section" of Form DC-2. Inserted documents are recorded in the "Other Records" section of Form DC-2.
- Consistency and completeness of the CSF are verified by the document control officer or his/her representative.
- Shipments of deliverable packages are documented by the document control officer or his/her representative.
- Deliverable packages are shipped by the document control officer or his/her representative using custody seals in a manner such that opening the packages would break the seals.
- Custody seals are signed and dated by the document control officer or his/her representative before placing them on deliverable packages.

EXHIBIT G

GLOSSARY OF TERMS

GLOSSARY OF TERMS

ABSORBANCE - a measure of the decrease in incident light passing through a sample into the detector. It is defined mathematically as:

$$A = \frac{I(\text{solvent})}{I(\text{solution})} = \log \frac{I_o}{I}$$

Where, I = radiation intensity

ALIQOT - a measured portion of a field sample taken for analysis.

ANALYSIS DATE/TIME - the date and military time (24-hour clock) of the introduction of the sample, standard, or blank into the analysis system.

ANALYTE - the element or ion an analysis seeks to determine; the element of interest.

ANALYTICAL SAMPLE - any solution or media introduced into an instrument on which an analysis is performed excluding instrument calibration, initial calibration verification, initial calibration blank, continuing calibration verification and continuing calibration blank. Note the following are all defined as analytical samples: undiluted and diluted samples (EPA and non-EPA), predigestion spike samples, duplicate samples, serial dilution samples, analytical spike samples, post-digestion spike samples, interference check samples (ICS), CRDL standard for AA (CRA), CRDL standard for ICP (CRI), laboratory control sample (LCS), preparation blank (PB) and linear range analysis sample (LRS).

ANALYTICAL SPIKE - the furnace spike at 2X CRDL or 20 ppb for lead added prior to analysis and after digestion, if digestion is required.

AUTOZERO - zeroing the instrument at the proper wavelength. It is equivalent to running a standard blank with the absorbance set at zero.

AVERAGE INTENSITY - the average of two different injections (exposures).

BACKGROUND CORRECTION - a technique to compensate for variable background contribution to the instrument signal in the determination of trace elements.

BATCH - a group of samples prepared at the same time in the same location using the same method.

CALIBRATION - the establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. The calibration standards must be prepared using the same type of acid or concentration of acids as used in the sample preparation.

CALIBRATION BLANK - a volume of acidified deionized/distilled water.

CALIBRATION STANDARDS - a series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).

CASE - a finite, usually predetermined number of samples collected over a given time period from a particular site. Case numbers are assigned by the Sample Management Office. A Case consists of one or more Sample Delivery Groups.

COEFFICIENT OF VARIATION (CV) - the standard deviation as a percent of the arithmetic mean.

CONCENTRATION LEVEL (low or medium) - for inorganics analysis, low or medium level is defined by the appropriate designation checked by the sampler on the Traffic Report.

CONTINUING CALIBRATION - analytical standard run every 10 analytical samples or every 2 hours, whichever is more frequent, to verify the calibration of the analytical system.

CONTRACT REQUIRED DETECTION LIMIT (CRDL) - minimum level of detection acceptable under the contract Statement of Work.

CONTROL LIMITS - a range within which specified measurement results must fall to be compliant. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that noncompliant data be flagged.

CORRELATION COEFFICIENT - a number (r) which indicates the degree of dependence between two variables (concentration - absorbance). The more dependent they are the closer the value to one. Determined on the basis of the least squares line.

DAY - unless otherwise specified, day shall mean calendar day.

DIGESTION LOG - an official record of the sample preparation (digestion).

DISSOLVED METALS - analyte elements in an aqueous sample which will pass through a 0.45 um filter.

DRY WEIGHT - the weight of a sample based on percent solids. The weight after drying in an oven.

DUPLICATE - a second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

FIELD BLANK - this is any sample that is submitted from the field and is identified as a blank. This includes trip blanks, rinsates, equipment blanks, etc.

FIELD SAMPLE - a portion of material received to be analyzed that is contained in single or multiple containers and identified by a unique EPA Sample Number.

FLAME ATOMIC ABSORPTION (AA) - atomic absorption which utilizes flame for excitation.

GRAPHITE FURNACE ATOMIC ABSORPTION (GFAA) - atomic absorption which utilizes a graphite cell for excitation.

HOLDING TIME - the elapsed time expressed in days from the date of receipt of the sample by the Contractor until the date of its analysis.

Holding time = (sample analysis date - sample receipt date)

INDEPENDENT STANDARD - a Contractor-prepared standard solution that is composed of analytes from a different source than those used in the standards for the initial calibration.

INDUCTIVELY COUPLED PLASMA (ICP) - a technique for the simultaneous or sequential multi-element determination of elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Characteristic atomic line emission spectra are produced by excitation of the sample in a radio frequency inductively coupled plasma.

IN-HOUSE - at the Contractor's facility.

INJECTION - introduction of the analytical sample into the instrument excitation system for the purpose of measuring absorbance, emission or concentration of an analyte. May also be referred to as exposure.

INSTRUMENT CALIBRATION - analysis of analytical standards for a series of different specified concentrations; used to define the quantitative response, linearity, and dynamic range of the instrument to target analytes.

INSTRUMENT DETECTION LIMIT (IDL) - determined by multiplying by three the standard deviation obtained for the analysis of a standard solution (each analyte in reagent water) at a concentration of 3x-5x IDL on three nonconsecutive days with seven consecutive measurements per day.

INSTRUMENT CHECK SAMPLE - a solution containing both interfering and analyte elements of known concentration that can be used to verify background and interelement correction factors.

INSTRUMENT CHECK STANDARD - a multi-element standard of known concentrations prepared by the analyst to monitor and verify instrument performance on a daily basis.

INTERFERENTS - substances which affect the analysis for the element of interest.

INTERNAL STANDARDS - in-house compounds added at a known concentration.

LABORATORY - synonymous with Contractor as used herein.

LABORATORY CONTROL SAMPLE (LCS) - a control sample of known composition. Aqueous and solid laboratory control samples are analyzed using the same sample preparation, reagents, and analytical methods employed for the EPA samples received.

LABORATORY RECEIPT DATE - the date on which a sample is received at the Contractor's facility, as recorded on the shipper's delivery receipt and Sample Traffic Report. Also referred to as VTSR (validated time of sample receipt).

LINEAR RANGE, LINEAR DYNAMIC RANGE - the concentration range over which the ICP analytical curve remains linear.

MATRIX - the predominant material of which the sample to be analyzed is composed. For the purpose of this SOW, a sample matrix is either water or soil/sediment. Matrix is not synonymous with phase (liquid or solid).

MATRIX MODIFIER - salts used in AA to lessen the effects of chemical interferents, viscosity, and surface tension.

MATRIX SPIKE - aliquot of a sample (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

METHOD OF STANDARD ADDITIONS (MSA) - the addition of 3 increments of a standard solution (spikes) to sample aliquots of the same size. Measurements are made on the original and after each addition. The slope, x-intercept and y-intercept are determined by least-square analysis. The analyte concentration is determined by the absolute value of the x-intercept. Ideally, the spike volume is low relative to the sample volume (approximately 10% of the volume). Standard addition may counteract matrix effects; it will not counteract spectral effects. Also referred to as Standard Addition.

PERCENT SOLIDS - the proportion of solid in a soil sample determined by drying an aliquot of the sample.

PERFORMANCE EVALUATION (PE) SAMPLE - a sample of known composition provided by EPA for Contractor analysis. Used by EPA to evaluate Contractor performance.

POST-DIGESTION SPIKE - the addition of a known amount of standard after digestion.

PREPARATION BLANK (reagent blank, method blank) - an analytical control that contains distilled, deionized water and reagents, which is carried through the entire analytical procedure (digested and analyzed). An aqueous method blank is treated with the same reagents as a sample with a water matrix; a solid method blank is treated with the same reagents as a soil sample.

PROTOCOL - a compilation of the procedures to be followed with respect to sample receipt and handling, analytical methods, data reporting and deliverables, and document control. Used synonymously with Statement of Work (SOW).

QUALITY CONTROL SAMPLE - a solution obtained from an outside source having known concentration values to be used to verify the calibration standards.

REAGENT BLANK - a volume of deionized, distilled water containing the same acid matrix as the calibration standards carried through the entire analytical scheme.

ROUNDING RULES - If the figure following those to be retained is less than 5, the figure is dropped, and the retained figures are kept unchanged. As an example, 11.443 is rounded off to 11.44.

If the figure following those to be retained is greater than 5, the figure is dropped, and the last retained figure is raised by 1. As an example, 11.446 is rounded off to 11.45.

If the figure following those to be retained is 5, and if there are no figures other than zeros beyond the five, the figure 5 is dropped, and the last-place figure retained is increased by one if it is an odd number or it is kept unchanged if an even number. As an example, 11.435 is rounded off to 11.44, while 11.425 is rounded off to 11.42.

If a series of multiple operations is to be performed (add, subtract, divide, multiply), all figures are carried through the calculations. Then the final answer is rounded to the proper number of significant figures.

See forms instructions (Exhibit B) for exceptions.

RUN - a continuous analytical sequence consisting of prepared samples and all associated quality assurance measurements as required by the contract Statement of Work.

SAMPLE DELIVERY GROUP (SDG) - a unit within a sample Case that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer samples within a Case, received over a period of up to 14 calendar days. Data from all samples in an SDG are due concurrently. A Sample Delivery Group is defined by one of the following, whichever occurs first:

- Case; or
- Each 20 samples within a Case; or
- Each 14-day calendar period during which samples in a Case are received, beginning with receipt of the first sample in the Case or SDG (seven calendar day period for 14-day data turnaround contracts).

Samples may be assigned to Sample Delivery Groups by matrix (i.e., all soils in one SDG, all waters in another), at the discretion of the laboratory.

SAMPLE NUMBER (EPA SAMPLE NUMBER) - a unique identification number designated by EPA for each sample. The EPA Sample Number appears on the sample Traffic Report which documents information on that sample.

SENSITIVITY - the slope of the analytical curve, i.e., functional relationship between emission intensity and concentration.

SERIAL DILUTION - the dilution of a sample by a factor of five. When corrected by the dilution factor, the diluted sample must agree with the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferences.

SOIL - synonymous with soil/sediment or sediment as used herein.

STOCK SOLUTION - a standard solution which can be diluted to derive other standards.

SUSPENDED - those elements which are retained by a 0.45 um membrane filter.

TOTAL METALS - analyte elements which have been digested prior to analysis.

TRAFFIC REPORT (TR) - an EPA sample identification form filled out by the sampler, which accompanies the sample during shipment to the laboratory and which is used for documenting sample condition and receipt by the laboratory.

VALIDATED TIME OF SAMPLE RECEIPT (VTSR) - the date on which a sample is received at the Contractor's facility, as recorded on the shipper's delivery receipt and Sample Traffic Report.

WET WEIGHT - the weight of a sample aliquot including moisture (undried).

10% FREQUENCY - a frequency specification during an analytical sequence allowing for no more than 10 analytical samples between required calibration verification measurements, as specified by the contract Statement of Work.

EXHIBIT H

DATA DICTIONARY AND FORMAT FOR DATA
DELIVERABLES IN COMPUTER-READABLE FORMAT

AGENCY STANDARD IMPLEMENTATION FOR INORGANICS ILM04.0

1. Format Characteristics

- 1.1 This constitutes an implementation of the EPA Agency Standard for Electronic Data Transmission based upon analytical results and ancillary information required by the contract. All data generated by a single analysis are grouped together, and the groups are aggregated to produce files that report data from an SDG. Because this implementation is only a subset of the Agency Standard, some fields have been replaced by delimiters as place holders for non-CLP data elements.
- 1.2 This implementation includes detailed specifications for the required format of each record. The position in the record where each field is to be contained relevant to other fields is specified, as well as the maximum length of the field. Each field's required contents are specified as literal (contained in quotes), which must appear exactly as shown (without quotes), or as a variable for which format and/or descriptions are listed in the format/contents column. Options and examples are listed for most fields. For fields where more than three options are available, a list and description of options are supplied following the record descriptions. Fields are separated from each other by the delimiter "|" (ASCII 124). Fields that do not contain data should be zero length with the delimiter as place holder.
- 1.3 Numeric fields may contain numeric digits, a decimal place, and a leading minus sign. A positive sign is assumed if no negative sign is entered in a numeric field and must not be entered into any numeric field.

Requirements for significant figures and number of decimal places are specified in Exhibit B. The numeric field lengths are specified such that all possible numeric values can be written to the file. The size of the numeric field indicates the maximum number of digits, decimal, and negative sign, if appropriate, that can appear in the field at the same time. Therefore, the number reported may need to be rounded (using EPA Rounding Rules) to fit into the field. The rounding must maintain the greatest significance possible providing the field length limitation. In addition, the rounded number that appears on the form, and therefore the field in the diskette file, must be used in any calculation that may result in other numbers reported on the same form or other forms in the SDG. Field lengths should only be as long as necessary to contain the data; packing with blanks is not allowed.

2. Record Types

- 2.1 The Agency Standard consists of variable length ASCII records. Maximum field length specifications match the reporting requirements in Exhibit B. The last two bytes of each record must contain "carriage return" and "line feed", respectively.
- 2.2 There are four groups of record types in the reporting format, as shown in this section. Detailed record formats follow.

Type	Type ID	Contents
Run Header	10	Information pertinent to a group of samples processed in a continuous sequence; usually several per SDG
Sample Header	20	Sample identifying, qualifying, and linking information
Results Record	30	Analyte results and qualifications
Comments Record	90	Free form comments

- 2.3 All record types given are mandatory. Type 10, representing the analytical run, contains the instrument and run IDs which act as an identifying label for the run. All 10, 20, 30, and 90 series records following that record pertain to the same analytical run. Type 20, representing the sample, contains the EPA Sample ID which acts as an identifying label for the sample. The QC code indicates whether the data is from an environmental sample, calibration, or QC sample. All 20, 30, and 90 series records following that record pertain to the same sample. Type 30, representing an individual analyte, contains an identifier to identify the analyte. All 30 series records following that record pertain to the same analyte.

3. Production Runs

A production run represents a "group" or "batch" of samples that are processed in a continuous sequence under relatively stable conditions. Specifically:

Calibration - All samples in a run use the same initial calibration data.

Method - Constant.

Instrument conditions - Constant throughout a run. Results obtained on different instruments cannot be combined in one run.

Thus, each separate group of analyses on each instrument will consist of a separate production run, and must be reported in a separate file.

The run numbers in an SDG must be unique; that is, there shall only be one Run Number "1", only one Run Number "2", etc. in an SDG.

In addition, later runs within a method for an analyte shall have a higher run number than earlier ones. For example, if arsenic is quantitated by the GFAA method on 01/01/94 beginning at 12:02 and arsenic is later quantitated by the GFAA method on 01/01/94 beginning at 18:06, then the run beginning at 12:02 shall have a lower run number than the run beginning at 18:06.

Example of the Sequence of Record Types in a Production Run

10 Contains run header information. Occurs once per run.

16 Contains additional run header information. Occurs once per run.

- 20 Acts as a header for the following instrument parameter information. Occurs once per run with EPA Sample Number equal to "IDL". Analysis year, analysis month, analysis day equal the year, month and day the IDLs were computed. Analyte count equals the number of the type 30 records that follow.
- 30 Contains only the Analyte CAS Number, IDL Label and IDL. Occurs once for each analyte used in the run.
 - 30
 - 30
 - 30
- 20 Acts as a header for the following instrument parameter information. Occurs once per run with EPA Sample Number equal to "LRV". Analysis year, analysis month, analysis day equal the year, month and day the linear ranges were computed. Analyte count equals the number of type 30, 32 and 34 groups that follow.
- 30 Contains only the Analyte CAS Number and the Analyte Identifier. Occurs once for each analyte used in the run.
 - 32 Contains integration time information for the preceding analyte on the type 30 record.
 - 34 Contains the CRDL and Linear Range information for the preceding analyte on the type 30 record. There are as many consecutive type 34 records as there are different wavelengths used for the analyte identified on preceding type 30.
 - 30
 - 32
 - 34
- 20 Acts as a header for the following instrument parameter information. Occurs once per run with EPA Sample Number equal to "BCD". Analysis year, analysis month, analysis day equal the year, month and day the background correction factors were computed. Analyte count equals the number of the type 30 and 35 groups that follow.
- 30 Contains only the Analyte CAS Number. Occurs once for each analyte used in the run.
 - 35 Contains the background and interelement correction information for the preceding analyte on the type 30 record. There are as many consecutive type 35 records as there are interelement correction factors for the analyte identified on preceding type 30.

30

35

20 Contains header information for sample and QC data.

21 Contains additional information for analytical and instrument QC samples. Will always be preceded by a type 20 record.

22 Contains additional information for analytical samples. Will usually follow type 21 record.

30 Contains the sample level concentration, true or added value and QC value for each analyte. Occurs once for each analytical result for the EPA Sample Number of the previous type 20 record.

31 Reports any instrumental data necessary to obtain the result reported on the previous type 30 record. Will always be preceded by a type 30 record. Occurs once per type 30 record.

30 Values for the next analyte wavelength being measured.

31 Values for the next analyte wavelength being measured.

30

31

Type 30-31 record sequence continues as many times as the value of the ANALYTE COUNT on the previous type 20 record.

20 Next Sample Header record - The following applies to the next sample data.

21

22

30

31

30

31 etc.

20

21

22

30

4. Record Sequence

- 4.1 A Run Header (type 10) record must be present as the first record in the file (run). Further occurrences of the type 10 record in the file are not allowed.

A type 16 record must immediately follow the type 10 record. Further occurrences of the type 16 record in the file are not allowed.

The first three type 20 records are headers for the run-wide instrument parameters.

The first type 20 record (followed by type 30 record[s] only) is a header for the quarterly determined and other instrument detection limit values (IDL) and must immediately follow the type 16 record.

The second type 20 record (of the type 30, 32, 34 group) is a header for the linear range values (LRV) and must immediately follow the last type 30 record that pertains to the instrument detection limit values. The linear range values for all methods except the ICP method are the analytically determined concentrations of the highest instrument calibration standards that are used in the generation of the calibration curve at the beginning of every run. The linear range values for the ICP method are the quarterly determined values that are reported on Form XII of the hardcopy.

The third type 20 record is a header for the ICP and GFAA background correction data (BCD) and must immediately follow the last type 34 record that pertains to the linear range values. This third type 20 record (of the type 30, 35 group) is not required for methods AV, CV, CA, AS and C (that is, mercury and cyanide analyses).

These are the only occurrences of the type 20 records that do not relate to actual analyses in the run. Therefore, the only fields that are not blank in these occurrences of the type 20 record are the RECORD TYPE ("20"); EPA SAMPLE NUMBER ("IDL", "LRV" and "BCD"); Analysis Year/Year Computed, Analysis Month/Month Computed, Analysis Day/Day Computed ("YY", "MM", "DD"); and ANALYTE COUNT.

A minimum of one type 30 record must immediately follow the first type 20 record, and the total number of type 30 records must be equivalent to the ANALYTE COUNT on this type 20 record.

A minimum of one group of type 30, 32 and 34 records must immediately follow the second type 20 record. The information in each group must pertain to one and only one analyte. The number of groups must be equivalent to the ANALYTE COUNT on the second type 20 record.

A minimum of one group of type 30 and 35 records must immediately follow the third type 20 record for background correction data (if required). The information in each group must pertain to one and only one analyte. The number of groups must be equivalent to the ANALYTE COUNT on the third type 20 record.

The type 20 record that relates to the analysis of the first instrument calibration standard must immediately follow the last type 30, 35 group for methods ICP and GFAA, or the last type 30, 32, 34 group for the methods for mercury and cyanide analyses. After the appearance of this type 20 record in the file, further occurrences of the type 32, 34 and 35 records in that file are not allowed.

- 4.2 Each environmental sample, calibration, or quality control sample is represented by a group composed of type 20, 21, and 22 records, which hold sample level identifying information, followed by a minimum of one group composed of type 30 and 31 records for each analyte's wavelength.

The type 20 record holds a count for the number of analyte wavelengths being used to determine results. The ANALYTE COUNTER must have a value equivalent to the number of type 30 groups associated with each type 20 record.

Except for the first three type 20 records for methods ICP and GFAA, and the first two type 20 records for the methods for mercury and cyanide analyses, all type 20 records should occur in the order of sample analysis.

- 4.3 Type 90 comment records may be defined to occupy any position except before the type 10 (header) record. Comments pertaining to the whole run such as ones on Cover Page must appear before the first type 20 record. Comments pertaining to a particular sample such as ones on Form I must appear after the type 20 record for that sample, but before the first type 30 record associated with that sample. Comments pertaining to a particular analyte or wavelength must appear after the type 30 record of that wavelength, but before the type 30 record of the following wavelength.
- 4.4 The type 92 record which contains the sample associated data that is reported at the bottom of Form I must appear anywhere after the type 22 record for that EPA FIELD SAMPLE, but before the type 20 record of the next sample.

5. File/Record Integrity

All record types must contain the following check fields to ensure file and record integrity:

Record Position	Field Length	Contents	Remarks
First Field	2	Record type or identifier	"10" or as appropriate
Last Field	5	Record sequence number	00000-99999, repeated as necessary
	4	Record checksum	Four hexadecimal digits ¹
	2	Contains CR and LF	

6. Dates and Times

Date or time-of-day information consists of successive groups of two decimal digits, each separated by delimiters. Dates are given in the order YY MM DD, and times as HH MM. All hours must be given as 00 to 23 using a 24 hour clock and must be local time.

7. Multiple Volume Data

There is no requirement under this format that all the data from an entire SDG fit onto a single diskette. However, each single production run must fit onto a single diskette if possible. If that is not possible, then it is necessary that all files start with a type 10 record, and that the multiple type 10 records for each file of the same production run be identical. Information for a single sample may not be split between files.

8. Deliverable

- 8.1 The file or files must be submitted on a 5-1/4 inch floppy diskette, which may be either a double-sided, double-density, 360 K-byte or a high capacity 1.2 M-byte, or 3.5 inch double-sided, double-density 720 K-byte or 1.44 M-byte, diskette. The diskette must be formatted and recorded using the MS-DOS Operating System. The diskette or diskettes must contain all information relevant to one and only one SDG, and must accompany the hardcopy package for the SDG submitted to the Sample Management Office (see Exhibit B). Information on the diskette or diskettes must correspond exactly with information submitted in the hardcopy data package and on the hardcopy data package forms. Blank or unused records should not be included on the diskettes.

¹The checksum is defined to be the sum of the ASCII representation of the data on the record up to the Record Sequence Number plus the checksum of the previous record. The sum is taken modulo 65536 (2^{16}) and represented as four (4) hexadecimal digits.

- 8.2 Each diskette must be identified with an external label containing (in this order) the following information:

Disk Density
File Name(s)
Laboratory Name (optional)
Laboratory Code
Case Number (where applicable)
SAS Number (where applicable)
Contract Number

The format for the File Name(s) must be XXXXXX.I01 to XXXXXX.I99

where XXXXXX is the SDG identifier, I designates inorganics, and 01 through 99 the file number.

Dimensions of the label must be in the range 4-3/4" to 5" long by 1 1/4" to 1 1/2" wide for 5 1/4 inch floppy diskette; and 2" to 2 1/4" long by 2 1/8" to 2 3/8" wide for 3.5 inch IBM-compatible diskette.

9. Record Listing

Following is a listing of every record type required to report data from a single SDG.

FORMAT OF THE PRODUCTION RUN FIRST HEADER RECORD (TYPE 10)

<u>MAXIMUM LENGTH</u>	<u>CONTENTS</u>	<u>FORMAT/CONTENTS</u>
2	RECORD TYPE	"10"
1	Delimiter	
2	ANALYSIS START YEAR	YY
1	Delimiter	
2	ANALYSIS START MONTH	MM
1	Delimiter	
2	ANALYSIS START DAY	DD
1	Delimiter	
2	ANALYSIS START HOUR	HH
1	Delimiter	
2	ANALYSIS START MINUTE	MM
1	Delimiter	
5	METHOD TYPE	CHARACTER ²
1	Delimiter	
8	METHOD NUMBER	"ILM04.0" (SOW)
1	Delimiter	
3	MANAGER'S INITIALS	CHARACTER
1	Delimiter	
6	LAB CODE	CHARACTER
4	Delimiter	
11	CONTRACT NUMBER	CHARACTER
1	Delimiter	
10	INSTRUMENT ID	CHARACTER
2	Delimiter	
25	LABORATORY NAME	CHARACTER
1	Delimiter	
2	RUN NUMBER	NUMERIC ³
1	Delimiter	
5	RECORD SEQUENCE NUMBER	NUMERIC
4	CHECKSUM	CHARACTER

²Method Types are

"P" for ICP

"A" for Flame AA

"F" for Furnace AA

"PM" for ICP when Microwave Digestion is used

"AM" for Flame AA when Microwave Digestion is used

"FM" for Furnace AA when Microwave Digestion is used

"CV" for Manual Cold Vapor AA

"AV" for Automated Cold Vapor AA

"CA" for Midi-Distillation Spectrophotometric

"AS" for Semi-Automated Spectrophotometric

"C" for Manual Spectrophotometric

"T" for Titrimetric

³Run number values are 01 through 99. Each production run will be assigned a unique Run Number. Run Numbers are to be assigned sequentially beginning with 01 and will equal the number of production runs.

FORMAT OF THE PRODUCTION RUN SECOND HEADER RECORD (TYPE 16)

<u>MAXIMUM LENGTH</u>	<u>CONTENTS</u>	<u>FORMAT/CONTENTS</u>
2	RECORD TYPE	"16"
1	Delimiter	
2	ANALYSIS END YEAR	YY
1	Delimiter	
2	ANALYSIS END MONTH	MM
1	Delimiter	
2	ANALYSIS END DAY	DD
1	Delimiter	
2	ANALYSIS END HOUR	HH
1	Delimiter	
2	ANALYSIS END MINUTE	MM
1	Delimiter	
1	AUTO-SAMPLER USED	"Y" or "N" ⁴
1	Delimiter	
1	INTERELEMENT CORRECTIONS APPLIED	"Y" or "N" ⁵
1	Delimiter	
1	BACKGROUND CORRECTIONS APPLIED	"Y" or "N" ⁵
1	Delimiter	
1	RAW DATA GENERATED	"Y" or "N" or "B" ⁶
1	Delimiter	
5	RECORD SEQUENCE NUMBER	NUMERIC
4	CHECKSUM	CHARACTER

⁴Enter "Y" if an auto-sampler is used with equal time and intervals between analysis.

⁵These are the answers to the first two questions on the Cover Page. "Y" equals "YES", and "N" equals "NO".

⁶This is the answer to the third question on the Cover Page. "Y" equals "YES", "B" equals BLANK and "N" equals "NO".

FORMAT FOR THE MANDATORY SAMPLE HEADER DATA RECORD (TYPE 20)

<u>MAXIMUM LENGTH</u>	<u>CONTENTS</u>	<u>FORMAT/CONTENTS</u>
2	RECORD TYPE	"20"
1	Delimiter	
2	REGION	NUMERIC
1	Delimiter	
12	EPA SAMPLE NUMBER	CHARACTER ⁷
1	Delimiter	
5	MATRIX	CHARACTER ⁸
1	Delimiter	
3	QC CODE	CHARACTER
1	Delimiter	
3	SAMPLE QUALIFIER	CHARACTER ⁹
1	Delimiter	
5	CASE NUMBER	CHARACTER
1	Delimiter	
6	SDG NUMBER	CHARACTER
1	Delimiter	
2	ANALYSIS YEAR/YEAR COMPUTED	YY
1	Delimiter	
2	ANALYSIS MONTH/MONTH COMPUTED	MM
1	Delimiter	
2	ANALYSIS DAY/DAY COMPUTED	DD
1	Delimiter	
2	ANALYSIS HOUR	HH
1	Delimiter	
2	ANALYSIS MINUTE	MM
2	Delimiter	
2	SAMPLE WT/VOL UNITS	"G" / "ML" ¹⁰
1	Delimiter	
5	SAMPLE WT/VOL	NUMERIC ¹¹
1	Delimiter	
3	ANALYTE COUNT	NUMERIC
1	Delimiter	
5	RECORD SEQUENCE NUMBER	NUMERIC
4	CHECKSUM	CHARACTER

⁷EPA Sample Number as appears on Form XIV except for the first three type 20 records. The first type 20 record must have an EPA Sample Number of "IDL"; the second, an EPA sample number of "LRV"; the third, an EPA sample number of "BCD".

⁸For matrix, "1" equals "WATER", and "F" equals "SOIL".

⁹"REJ" sample qualifier is for the unacceptable (one of the two) MSA results; this sample qualifier appears on the type 20 record containing the zero (0) addition EPA Sample Number (XXXXXX0).

¹⁰"G" equals grams, and "ML" equals milliliters.

¹¹This is the size of the sample at the beginning of the digestion procedure.

SAMPLE QC CODES LISTING FOR TYPE 20

NOTE: These QC codes appear in the QC code fields on type 20 records. They are used to indicate the type of data that is being reported.

<u>QCC</u>	<u>Name</u>	<u>Definition</u>
LRB	LABORATORY (REAGENT) BLANK	The Preparation or Method Blank (See Exhibit G).
LCB	LABORATORY CALIBRATION BLANK	The Continuing Calibration Blank (CCB) (See Exhibit G).
LIB	LABORATORY INITIAL BLANK	The Initial Calibration Blank (ICB) (See Exhibit G).

LCM	LABORATORY CONTROL SOLUTION	The Laboratory Control Sample (LCS) (See Exhibit G).

LD1	LABORATORY DUPLICATE FIRST MEMBER	This is the same as the Sample Result "(S)" that is reported on the Duplicate Form of hardcopy (Form VI).
LD2	LABORATORY DUPLICATE SECOND MEMBER	This is the second aliquot and is identified as "D" on the Duplicate Form of hardcopy (Form VI).

LVM	LABORATORY CALIBRATION VERIFICATION SOLUTION	These values are identified as "Initial Calibration Verification" (ICV) on Form II (Part 1).
LVC	LABORATORY CONTINUING CALIBRATION VERIFICATION	These values are identified as "Continuing Calibration Verification" (CCV) on Form II (Part 1).

LSO	LABORATORY SPIKED SAMPLE BACKGROUND (ORIGINAL) VALUES	These values are identified as "Sample Result (SR)" on the "Spike Sample Recovery" Form of hardcopy (Form V (Part 1)).
LSF	LABORATORY SPIKED SAMPLE-FINAL VALUES	These are the "Spiked Sample Result (SSR)" values on the "Spike Sample Recovery" Form of hardcopy (Form V (Part 1)).

LDO	LABORATORY DILUTED SAMPLE BACKGROUND (ORIGINAL) VALUES	These values are the "Initial Sample Result (I)" values on the "Serial Dilution" Form of hardcopy (Form IX).
LDF	LABORATORY DILUTED SAMPLE - FINAL VALUES	These are the "Serial Dilution Result(S)" values on the "Serial Dilution" Form of hardcopy (Form IX).

SAMPLE QC CODES LISTING FOR TYPE 20

<u>OCC</u>	<u>Name</u>	<u>Definition</u>
MS0	STANDARD ADDITION RESULTS ORIGINAL VALUE	This value is identified as "0 ADD" on "Standard Addition Results", Form VIII.
MS1	STANDARD ADDITION RESULTS FIRST ADDITION	This value is identified as "1 ADD" on "Standard Addition Results", Form VIII.
MS2	STANDARD ADDITION RESULTS SECOND ADDITION	This value is identified as "2 ADD" on "Standard Addition Results", Form VIII.
MS3	STANDARD ADDITION RESULTS THIRD ADDITION	This value is identified as "3 ADD" on "Standard Addition Results", Form VIII.
PDO	POST-DIGESTION SPIKE BACKGROUND (ORIGINAL) VALUES	This value is identified as "Sample Result" (SR) on the "Post Digest Spike Sample Recovery", Form V (Part 2).
PDF	POST-DIGESTION SPIKE BACKGROUND (FINAL) VALUES	This value is identified as "Spiked Sample Result" (SSR) on the "Post Digest Spike Sample Recovery", Form V (Part 2).
LPC	CRDL STANDARD	Laboratory Performance Check Solution for ICP (CRI) and Graphite Furnace (CRA).
LII	LABORATORY INTERFERENCE CHECK SOLUTION (INITIAL)	The results of this solution analysis are reported on the "Interference Check Sample" (ICS), Form IV.
LIF	LABORATORY INTERFERENCE CHECK SOLUTION (FINAL)	The results of this solution analysis are reported on the "Interference Check Sample" (ICS), Form IV.
FRB	FIELD BLANK	This is any sample that is submitted from the field and is identified as a blank. This includes trip blanks, rinsates, equipment blanks, etc.

FORMAT OF THE SAMPLE HEADER RECORD (TYPE 21)

<u>MAXIMUM LENGTH</u>	<u>CONTENTS</u>	<u>FORMAT/CONTENTS</u>
2	RECORD TYPE	"21"
2	Delimiter	
3	LEVEL	"LOW"/"MED"
3	Delimiter	
6	SAS NUMBER	CHARACTER
1	Delimiter	
14	LAB SAMPLE ID	CHARACTER
1	Delimiter	
2	PREPARATION YEAR	YY
1	Delimiter	
2	PREPARATION MONTH	MM
1	Delimiter	
2	PREPARATION DAY	DD
2	Delimiter	
2	YEAR RECEIVED	YY
1	Delimiter	
2	MONTH RECEIVED	MM
1	Delimiter	
2	DAY RECEIVED	DD
1	Delimiter	
9	SOLUTION SOURCE	CHARACTER ¹²
1	Delimiter	
8	INJECTION/ALIQOT VOLUME	NUMERIC ¹³
1	Delimiter	
2	PREPARATION START HOUR	HH ¹⁴
1	Delimiter	
5	RECORD SEQUENCE NUMBER	NUMERIC
4	CHECKSUM	CHARACTER

¹²This is the source of the solutions that is reported on Forms IIA, IIB, IV and VII of the hardcopy (ICV, CCV, CRI, CRA, ICS, and LCS).

¹³This is the portion of the sample that is injected into the instrument excitation system for the purpose of measuring the absorbance, emission or concentration of an analyte.

¹⁴This is the hour at which the preparation is started. It is used to differentiate between different batches on the same day.

FORMAT OF THE ASSOCIATED INJECTION AND COUNTER RECORD (TYPE 22)

<u>MAXIMUM LENGTH</u>	<u>CONTENTS</u>	<u>FORMAT/CONTENTS</u>
2	RECORD TYPE	"22"
10	Delimiter	
8	FINAL VOLUME	NUMERIC ¹⁵
1	Delimiter	
8	DILUTION FACTOR	NUMERIC
3	Delimiter	
5	PERCENT SOLIDS	NUMERIC
1	Delimiter	
5	RECORD SEQUENCE NUMBER	NUMERIC
4	CHECKSUM	CHARACTER

¹⁵This is the final volume that is currently reported on Form XIII of the hardcopy.

FORMAT OF THE RESULTS DATA RECORD (TYPE 30)

<u>MAXIMUM LENGTH</u>	<u>CONTENTS</u>	<u>FORMAT/CONTENTS</u>
2	RECORD TYPE	"30"
1	Delimiter	
1	ANALYTE IDENTIFIER	"C"/"I" ¹⁶
1	Delimiter	
9	ANALYTE CAS NUMBER	CHARACTER
2	Delimiter	
5	CONCENTRATION UNITS	"UG/L"/"MG/KG"
1	Delimiter	
3	CONCENTRATION QUALIFIER	CHARACTER ¹⁷
1	Delimiter	
15	CONCENTRATION	NUMERIC ^{18,19,20}
1	Delimiter	
1	VALUE DESCRIPTOR	"T"/"F" ²¹
1	Delimiter	
10	AMOUNT ADDED OR TRUE VALUE	NUMERIC
1	Delimiter	
1	QC VALUE DESCRIPTOR, P	"P" ²²
1	Delimiter	
10	QC VALUE	NUMERIC
1	Delimiter	
1	QC VALUE DESCRIPTOR, C	"C" ²²
1	Delimiter	
10	QC VALUE	NUMERIC
1	Delimiter	
1	QC VALUE DESCRIPTOR, L	"L" ²²
1	Delimiter	
10	QC VALUE	NUMERIC
1	Delimiter	
1	MATRIX SPIKE QC LIMIT QUALIFIER	"N" ²³
1	Delimiter	
10	QC LOWER LIMIT	NUMERIC ²⁴
1	Delimiter	
10	QC UPPER LIMIT	NUMERIC ²⁴
1	Delimiter	
1	QC LIMIT QUALIFIER	"*"/"E" ²⁵
1	Delimiter	
1	IDL LABEL	"U"
1	Delimiter	
10	IDL	NUMERIC ²⁶
1	Delimiter	
1	RAW DATA AVERAGE QUALIFIER	"U"/"B"/"L" ²⁷
1	Delimiter	
10	RAW DATA AVERAGE	NUMERIC ²⁸
1	Delimiter	
1	RAW DATA %RSD QUALIFIER	"M"/BLANK ²⁹
1	Delimiter	
10	RAW DATA %RSD	NUMERIC
1	Delimiter	
1	"MSA-TREE" QUALIFIER	"+"/"E"/"W"/BLANK ³⁰
1	Delimiter	
5	RECORD SEQUENCE NO.	NUMERIC
4	CHECKSUM	CHARACTER

FORMAT OF THE RESULTS DATA RECORD (TYPE 30) FOOTNOTES

- 16 "C" (CAS Registry Number) is used for all analytes except cyanide. "I" is used for cyanide.
- 17 "BDL" means below detection limit.
- "NSQ" means there is not sufficient quantity to analyze sample according to the protocol.
- "NAI" not analyzed due to interference, "NAR" no analysis result required.
- "LTC" means less than the CRDL but greater than or equal to the IDL.
- "FQC" means failed quality control criteria.
- "GTL" means greater than the linear range.
- "RIN" means that the analysis result was not used to report data in the SDG. The results are reported from a later reanalysis of the same sample aliquot.
- "REX" means that the analysis result was not used to report data in the SDG. The results are reported from a later reanalysis of a repreparation of the same sample.
- Note that, except for "NAR", none of these codes relieves the Contractor from reporting a valid result. They only explain why or if the result is qualified.
- 18 The GFAA analytical or post-digestion spike sample result (SSR) must always be reported in ug/L; do not convert from ug/L to mg/Kg for soil samples. In addition, the GFAA post-digestion SSR shall not be corrected for dilutions.
- 19 EPA FIELD SAMPLES (Form I equivalents) that do not have QC codes shall have their analytes' results reported to four decimal places. Also, results for samples that carry the QC codes MS0 and FRB shall be reported to four decimal places.
- 20 Follow the instructions for the reporting of data in Exhibit B in reporting results for samples with QC codes. For example, the LD2 QC code sample results shall be reported to four decimal places because the duplicate result on Form VI has to be reported to four decimal places. Refer to pages H-13 and H-14 for QC codes and definitions.
- 21 "T" stands for a true value of the solution. This includes the concentration of all (ICP as well) instrument calibration standards. "F" stands for an added concentration to a sample such as a pre- or post-digestion spike, or MSA additions.
- 22 "P" equals percent recovery (%R), percent difference (%D), or relative percent difference (RPD), "C" equals MSA correlation coefficient, and "L" equals control limit for duplicates. For GFAA analysis, the EPA duplicate sample number with the "D" suffix should contain the RPD value, and the EPA duplicate sample number with the "DA" suffix should contain the post-digestion spike sample %R value.

- 23 "N" is the qualifier that is used on Form V (Part 1) of the hardcopy to indicate that the matrix or pre-digestion spike sample recovery for an analyte is not within the specified control limits.
- 24 These are the limits for the spike sample recovery (Form VA), the ICV/CCV (Form IIA), the CRA/CRI (Form IIB), the ICSAB (Form IV), the LCS (Form VII), and the GFAA post-digestion spike recovery.
- 25 "*" is the qualifier that is used on Form VI of the hardcopy to indicate that the duplicate sample analysis for an analyte is out of control, and "E" is the qualifier that is used on Form IX of the hardcopy to indicate that the ICP serial dilution analysis results are estimated because of the existence of significant physical or chemical interferences. The "*" qualifier should be entered on the type 30 record of the EPA sample number with the "D" suffix; that is, on either the LD2 or MS0 (when duplicate result is quantitated by MSA) QC code type 30 record.
- 26 The IDL must be reported to one decimal place.
- 27 "U" means less than the IDL, "B" means less than the CRDL and greater than or equal to the IDL, "L" means greater than the linear range.
- 28 The average value of the replicate injections or exposures are reported in this field. The average values for mercury and cyanide analyses are also reported in this field.
- Exception: For MSA analysis, the single injection absorbance values are reported only in the "First Instrument Value" field of the type 31 record; do not report raw data average values for the single injection MSAs in the "Raw Data Average" field of the type 30 record. The "Raw Data Average" field of MS0 QC code shall contain the value of the MSA minus x-intercept; this value is also reported in the "Final Conc." column of Form VIII of the hardcopy.
- 29 "M" is the qualifier that is used to indicate that the replicate injection readings of the GFAA sample analysis do not agree within 20% relative standard deviation (RSD) or coefficient of variation (CV) for analytical samples.
- 30 "+" indicates that the MSA correlation coefficient is less than 0.995, "E" indicates that the GFAA post-digestion spike sample recovery (after dilution) is less than 40%, and "W" indicates that the GFAA post-digestion spike recovery is not within the recovery limits of 85-115% when three times the sample result is less than the spike sample result.

FORMAT FOR THE INSTRUMENTAL DATA READOUT (TYPE 31)

MAXIMUM LENGTH	CONTENTS	FORMAT/CONTENTS
2	RECORD TYPE	"31"
1	Delimiter	
1	TYPE OF DATA	"W" ³¹
1	Delimiter	
1	TYPE OF VALUE	CHARACTER ³²
2	Delimiter	
8	ANALYTE WAVELENGTH	NUMERIC (TO 2 DECIMAL PLACES)
1	Delimiter	
10	FIRST INSTRUMENT VALUE	NUMERIC ^{33,34}
2	Delimiter	
10	SECOND INSTRUMENT VALUE	NUMERIC ³³
2	Delimiter	
10	THIRD INSTRUMENT VALUE	NUMERIC ³³
2	Delimiter	
10	FOURTH INSTRUMENT VALUE	NUMERIC ³³
2	Delimiter	
10	FIFTH INSTRUMENT VALUE	NUMERIC ³³
1	Delimiter	
5	RECORD SEQUENCE NUMBER	NUMERIC
4	CHECKSUM	CHARACTER

³¹"W" equals wavelength.

³²"C" equals concentration in ug/L, "T" equals concentration in ug/250 ml, "F" equals concentration in ug/50 ml, "B" equals absorbance, "I" equals intensity, "A" equals peak area in cm square, and "H" equals peak height in cm.

³³This is used to report data for method analyses that require replicate injections or exposures. If a single instrument measurement is used, then enter it in the first instrument value field, and leave the other four fields empty. If two instrument measurements are used, then enter them in the first and second instrument value fields in the order of their analyses, and leave the other three fields empty; etc.

³⁴GFAA MSA analyses are single injections only. The EPA samples have the suffixes 0, 1, 2, and 3 (MAX123D0, MAX123D1, MAX123D2, MAX123D3), and their respective QC codes are MS0, MS1, MS2, and MS3. The absorbances for the four additions (zero, first, second, and third) shall be reported in this field, the first instrument value field. The -(x-intercept) concentration, which is also reported on the hardcopy of Form VIII in the "Final Conc" column, shall be reported in the "Raw Data Average" field of the MS0 QC code type 30 record. Therefore, do not report raw data averages (in the "Raw Data Average" field) for the MSA single injections on any of the four type 30 records. The absorbances of all four single injections shall only be reported in their respective type 31 record "First Instrument Value" fields. The MSA final concentration corrected for volume, sample weight, % solids, and dilution shall be reported in the "CONCENTRATION" field of the MS0 QC code type 30 record.

FORMAT OF THE AUXILIARY DATA RECORD (TYPE 32)

<u>MAXIMUM LENGTH</u>	<u>CONTENTS</u>	<u>FORMAT/CONTENTS</u>
2	RECORD TYPE	"32"
10	Delimiter	
2	INTEGRATION TIME CODE	"IT"
1	Delimiter	
10	INTEGRATION TIME	IN SECONDS
4	Delimiter	
5	RECORD SEQUENCE NUMBER	NUMERIC
4	CHECKSUM	CHARACTER

FORMAT OF THE QC LIMIT RECORD (TYPE 34)

<u>MAXIMUM LENGTH</u>	<u>CONTENTS</u>	<u>FORMAT/CONTENTS</u>
2	RECORD TYPE	"34"
4	Delimiter	
8	ANALYTE WAVELENGTH	NUMERIC (TO 2 DECIMAL PLACES)
1	Delimiter	
10	CRDL	NUMERIC
1	Delimiter	
10	LINEAR RANGE VALUE	NUMERIC
6	Delimiter	
5	RECORD SEQUENCE NO.	NUMERIC
4	CHECKSUM	CHARACTER

FORMAT OF THE CORRECTION DATA RECORD (TYPE 35)

<u>MAXIMUM LENGTH</u>	<u>CONTENTS</u>	<u>FORMAT/CONTENTS</u>
2	RECORD TYPE	"35"
1	Delimiter	
3	TYPE OF CORRECTION	"ICP"/"BG" ³⁵
1	Delimiter	
5	TYPE OF BACKGROUND	"BS"/"BD"/"BZ"
4	Delimiter	
9	CAS NUMBER OF INTERFERING ANALYTE	CHARACTER
1	Delimiter	
8	ANALYTE WAVELENGTH	NUMERIC (TO 2 DECIMAL PLACES)
1	Delimiter	
10	CORRECTION FACTOR	NUMERIC
1	Delimiter	
5	RECORD SEQUENCE NO.	NUMERIC
4	CHECKSUM	CHARACTER

³⁵"ICP" indicates interelement correction, while "BG" indicates a background correction.

FORMAT OF THE COMMENT RECORD (TYPE 90)

<u>MAXIMUM LENGTH</u>	<u>CONTENTS</u>	<u>FORMAT/CONTENTS</u>
2	RECORD TYPE	"90"
1	Delimiter	
67	ANY COMMENT	CHARACTER
1	Delimiter	
5	RECORD SEQUENCE NUMBER	NUMERIC
4	CHECKSUM	CHARACTER

FORMAT OF THE SAMPLE ASSOCIATED DATA RECORD (TYPE 92)

<u>MAXIMUM LENGTH</u>	<u>CONTENTS</u>	<u>FORMAT/CONTENTS</u>
2	RECORD TYPE	"92"
1	Delimiter	
9	COLOR BEFORE	CHARACTER
1	Delimiter	
9	COLOR AFTER	CHARACTER
1	Delimiter	
6	CLARITY BEFORE	CHARACTER
1	Delimiter	
6	CLARITY AFTER	CHARACTER
1	Delimiter	
6	TEXTURE	CHARACTER
1	Delimiter	
3	ARTIFACTS	"YES"/BLANK
1	Delimiter	
5	RECORD SEQUENCE NUMBER	NUMERIC
4	CHECKSUM	CHARACTER

APPENDIX A -- FORMAT OF RECORDS FOR SPECIFIC USES

DISCLAIMER

The USEPA does not warrant or guarantee the completeness and/or accuracy of the representative examples of record type uses provided in this appendix. This appendix serves as an example for the usage of record types and in no way redefines or supersedes the specifications or requirements stated in Exhibits A through H of ILM04.0.

Appendix A -- Format of Records for Specific Uses

Table of Contents

<u>Section</u>	<u>Page</u>
1.0 ICP	28
1.1 START OF AN ICP RUN WITH RECORD TYPES 10 & 16 AND THE FIRST THREE TYPE 20 RECORDS	28
1.2 ICP INSTRUMENT CALIBRATION STANDARDS, S0 AND S	29
1.3 SPIKE SAMPLE RECOVERY, DUPLICATES, AND SERIAL DILUTIONS PERFORMED ON THE SAME SAMPLE (QC CODES LSO & LSF, LD1 & LD2, LDO & LDF)	29
2.0 GFAA	32
2.1 START OF A GFAA RUN WITH RECORD TYPES 10 & 16 AND THE FIRST THREE TYPE 20 RECORDS	32
2.2 INSTRUMENT CALIBRATION STANDARDS BLANK (S0) & THREE OTHER STANDARDS	33
2.3 ANALYSIS OF A FIELD BLANK SAMPLE SAMPLE & ITS ANALYTICAL SPIKE SAMPLE WITH QC CODE FRB	34
2.4 SPIKE SAMPLE RECOVERY & DUPLICATES PERFORMED ON THE SAME SAMPLE (QC CODES LSO & LSF, AND LD1 & LD2)	34
2.5 DUPLICATES, WITH THE RESULT OF THE DUPLICATE SAMPLE QUANTITATED BY THE MSA (QC CODES LD1, LD2, MS0, MS1, MS2, MS3)	35
3.0 MERCURY (CVAA OR AVAA)	37
3.1 START OF A MERCURY RUN WITH RECORD TYPES 10 & 16 AND THE FIRST TWO TYPE 20 RECORDS	37
3.2 MERCURY INSTRUMENT CALIBRATION STANDARDS BLANK (S0) AND FOUR OTHER STANDARDS	37
3.3 SPIKE SAMPLE RECOVERY & DUPLICATES PERFORMED ON DIFFERENT SAMPLES (QC CODES LSO & LSF, AND LD1 & LD2)	38
3.4 SPIKE SAMPLE RECOVERY & DUPLICATES PERFORMED ON THE SAME SAMPLE (QC CODES LSO & LSF, AND LD1 & LD2)	38
3.5 INITIAL CALIBRATION VERIFICATION (ICV) WITH LVM QC CODE . . .	39
3.6 LABORATORY CONTROL SAMPLE (SOLID) WITH LCM QC CODE	39
4.0 CYANIDE (CA, AS, C, T)	40
4.1 START OF A CYANIDE RUN WITH RECORD TYPES 10 & 16 AND THE FIRST TWO TYPE 20 RECORDS	40
4.2 CYANIDE INSTRUMENT CALIBRATION STANDARDS BLANK (S0) AND FIVE OTHER STANDARDS	40
4.3 PREPARATION BLANK (SOIL) WITH LRB QC CODE	41
4.4 LABORATORY CONTROL SAMPLE (SOIL) WITH LCM QC CODE	41
4.5 CONTINUING CALIBRATION VERIFICATION (CCV) WITH LVC QC CODE .	41
4.6 SPIKE SAMPLE RECOVERY & DUPLICATES PERFORMED ON THE SAME SAMPLE (QC CODES LSO & LSF, AND LD1 & LD2)	41

1.0 ICP

1.1 START OF AN ICP RUN WITH RECORD TYPES 10 & 16 AND THE FIRST THREE TYPE 20 RECORDS

10|93|09|17|09|06|P|ILM04.0|ABC|TESLAB|||68-D2-0039|P2|
|TEST LABS INC.|2|000001879
16|93|09|17|12|03|Y|Y|Y|N|000012114

20|1|IDL|||93|07|15|||04|000044B9D
30|C|7440-22-4|||U|3.4|||000055996
30|C|7429-90-5|||U|22.8|||0000667D1
30|C|7440-39-3|||U|1.0|||0000775CB
30|C|7440-41-7|||U|0.4|||0000883C5

20|1|LRV|||93|07|15|||04|0002356C2
30|C|7440-22-4|||0002463D1
32|||IT|5.00|||000256CDA
34|||328.00|10|40000|||000267591
30|C|7429-90-5|||0002782AD
32|||IT|5.00|||000288BB6
34|||308.20|200|1000000|||0002994FB
30|C|7440-39-3|||00030A211
32|||IT|5.00|||00031AB1A
34|||493.40|200|100000|||00032B436
30|C|7440-41-7|||00033C149
32|||IT|5.00|||00034CA52
34|||313.00|5|25000|||00035D2DA

20|1|BCD|||93|07|01|||04|0007894FB
30|C|7440-22-4|||00079A20A
35|ICP|||7439-89-6|259.90|-0.0002500|00080AC9B
35|ICP|||7439-96-5|257.60|0.0002200|00081B6F4
30|C|7429-90-5|||00082C410
35|ICP|||7439-96-5|257.60|0.0004900|00083CE72
35|ICP|||7440-62-2|292.40|-0.0419200|00084D8EF
30|C|7440-39-3|||00085E605
35|ICP|||7439-96-5|257.60|0.0000600|00086F060
30|C|7440-41-7|||00087FD73
35|ICP|||7440-50-8|324.70|0.0046200|0008914D1
35|ICP|||7439-96-5|257.60|0.0015400|000901F30

1.2 ICP INSTRUMENT CALIBRATION STANDARDS, S0 AND S

```

20|1|S0|1|||20596|MAX123|93|09|17|09|06|||04|00128D199
21|||||STDB|||||TESLAB|||00129DD31
22|||||1.00|||00130E598
30|C|7440-22-4|||T|0.0|||U|3.4|0.0359|||00131F8F5
31|W|I||328.00|0.0304|0.0374|0.0400|||001320305
30|C|7429-90-5|||T|0.0|||U|22.8|0.0120|||001331697
31|W|I||308.20|0.0104|0.0136|0.0120|||001342137
30|C|7440-39-3|||T|0.0|||U|1.0|0.0000|||00135348D
31|W|I||493.40|-0.0002|0.0002|0.0000|||001363EA4
30|C|7440-41-7|||T|0.0|||U|0.4|0.0004|||0013751FA
31|W|I||313.00|0.0006|0.0002|0.0004|||001385C04

20|1|S|1|||20596|MAX123|93|09|17|09|11|||04|00206314E
21|||||STD1|||||TESLAB|||002073CD5
22|||||1.00|||00208453C
30|C|7440-39-3|||T|5000|||U|1.0|1.9603|||002139157
31|W|I||493.40|1.9540|1.9610|1.9660|||002149B6E
30|C|7440-41-7|||T|1000|||U|0.4|0.8401|||00215ADE2
31|W|I||313.00|0.8384|0.8378|0.8440|||00216B7EC
30|C|7440-43-9|||T|5000|||U|1.5|1.9951|||00219E77D
31|W|I||226.50|1.9460|1.9510|1.9684|||00220F18F
30|C|7440-48-4|||T|5000|||U|1.5|0.9948|||002210410
31|W|I||228.60|0.9924|0.9910|1.0010|||002220E25

```

1.3 SPIKE SAMPLE RECOVERY, DUPLICATES, AND SERIAL DILUTIONS PERFORMED ON THE SAME SAMPLE (QC CODES LSO & LSF, LD1 & LD2, LDO & LDF)

```

20|1|MAX123|F|LSO||20596|MAX123|93|09|17|11|09|G|1.05|08|01568C5FD
21|LOW|||S308233-01|93|09|14|93|08|24||8|01569D451
22|||||200|1.00||91.5|01570DE17
90|STONES|01571E154
92|GREY|GREY||MEDIUM|YES|01572EA43
30|C|7440-22-4|MG/KG|BDL|0.7078|||U|3.4|U|1.1600|||01573FD12
31|W|C||328.00|4.2000|0.5500|-1.2800|||0157409A5
30|C|7429-90-5|MG/KG|NAR|6227.0101|||U|22.8|29913.0000|||
|015751DCD
31|W|C||308.20|29992.0000|29654.0000|30093.0000|||015762CA0
30|C|7440-39-3|MG/KG|LTC|21.9349|||U|1.0|B|105.3700|||
|01577400C
31|W|C||493.40|107.2400|101.6400|107.2400|||015784DA6
30|C|7440-41-7|MG/KG|LTC|0.3102|||U|0.4|B|1.4900|||01579606A

```


31|W|C||313.00|1.4900||1.4900||1.4900|||015806CD9
 30|C|7440-70-2||MG/KG|NAR|682.2795|||U|35.7|B|3277.5000|||
 |0158180EE
 31|W|C||317.90|3289.9000||3259.6000||3283.1000|||015828F39
 30|C|7440-43-9||MG/KG|BDL|0.3123|||U|1.5|U|-0.2200|||
 |01583A22B
 31|W|C||226.50|-0.7600||0.8300||-0.7500|||01584AEF6
 30|C|7440-48-4||MG/KG|LTC|3.4161|||U|1.5|B|16.4100|||
 |01585C1F7
 31|W|C||228.60|14.7300||16.7400||17.7500|||01586CF0C
 30|C|7440-47-3||MG/KG||7.7398|||U|1.8||37.1800|||01587E103
 31|W|C||267.70|39.6500||36.8600||35.0200|||01588EE1F

 20|1|MAX123|F|LD1||20596|MAX123|93|09|17|11|09||G|1.05|08|016094756
 21||LOW|||S308233-01|93|09|14||93|08|24||8|0161055AA
 22|||200|1.00||91.5|016115F70
 30|C|7440-22-4||MG/KG|BDL|0.7078|||U|3.4|U|1.1600|||01573FD12
 31|W|C||328.00|4.2000||0.5500||-1.2800|||0157409A5
 30|C|7429-90-5||MG/KG||6227.0101|||U|22.8||29913.0000|||
 |015751DCD
 31|W|C||308.20|29992.0000||29654.0000||30093.0000|||015762CA0
 30|C|7440-39-3||MG/KG|LTC|21.9349|||U|1.0|B|105.3700|||
 |01577400C
 31|W|C||493.40|107.2400||101.6400||107.2400|||015784DA6
 30|C|7440-41-7||MG/KG|LTC|0.3102|||U|0.4|B|1.4900|||01579606A
 31|W|C||313.00|1.4900||1.4900||1.4900|||015806CD9
 30|C|7440-70-2||MG/KG|LTC|682.2795|||U|35.7|B|3277.5000|||
 |0158180EE
 31|W|C||317.90|3289.9000||3259.6000||3283.1000|||015828F39
 30|C|7440-43-9||MG/KG|BDL|0.3123|||U|1.5|U|-0.2200|||
 |01583A22B
 31|W|C||226.50|-0.7600||0.8300||-0.7500|||01584AEF6
 30|C|7440-48-4||MG/KG|LTC|3.4161|||U|1.5|B|16.4100|||
 |01585C1F7
 31|W|C||228.60|14.7300||16.7400||17.7500|||01586CF0C
 30|C|7440-47-3||MG/KG||7.7398|||U|1.8||37.1800|||01587E103
 31|W|C||267.70|39.6500||36.8600||35.0200|||01588EE1F
 20|1|MAX123|F|LDO||20596|MAX123|93|09|17|11|09||G|1.05|03|01650C630
 21||LOW|||S308233-01|93|09|14||93|08|24||8|01651D484
 22|||200|1.00||91.5|01652DE4A
 30|C|7440-22-4||UG/L|BDL|3.40|||U|3.4|U|1.1600|||01655FC98

31|W|C||328.00|4.2000||0.5500||-1.2800|||01656092B
 30|C|7429-90-5|UG/L|29913.00|||U|22.8||29913.0000|||
 |016571C09
 31|W|C||308.20|29992.0000||29654.0000||30093.0000|||016582ADC
 30|C|7440-39-3|UG/L|LTC|105.37|||U|1.0|B|105.3700|||
 |016593DCE
 31|W|C||493.40|107.2400||101.6400||107.2400|||016604B68

 20|1|MAX123D|F|LD2||20596|MAX123|93|09|17|11|11|G|1.04|08|016913BCF
 21||LOW|||S308233-02|93|09|14||93|08|24||8|016924A23
 22|||200|1.00||90.9|0169353EC
 30|C|7440-22-4|MG/KG|BDL|0.7146|||U|3.4|U|0.9600|||0169466BE
 31|W|C||328.00|1.6400||1.6300||-0.3800|||016957356
 30|C|7429-90-5|MG/KG|6622.7406||P|6.2|||U|22.8||31511.0000|||
 |016968784
 31|W|C||308.20|31993.0000||31313.0000||31226.0000|||016979641
 30|C|7440-39-3|MG/KG|LTC|25.1387||P|13.6|||U|1.0|B|119.6100|||
 |01698AAC5
 31|W|C||493.40|121.4600||118.9300||118.4300|||01699B86C
 30|C|7440-41-7|MG/KG|LTC|0.3153||P|1.6|||U|0.4|B|1.5000|||
 |01700CC13
 31|W|C||313.00|1.5000||1.5000||1.5000|||01701D86A
 30|C|7440-70-2|MG/KG|LTC|676.6709||P|0.8|||U|35.7|B|3219.6000|||
 |01702ED66
 31|W|C||317.90|3256.5000||3214.5000||3187.8000|||01703FBA7
 30|C|7440-43-9|MG/KG|BDL|0.3153|||U|1.5|U|-0.9400|||
 |017040EA5
 31|W|C||226.50|-0.3300||-0.7400||-1.7400|||017051B96
 30|C|7440-48-4|MG/KG|LTC|3.8714||P|12.5|||U|1.5|B|18.4200|||
 |017062FB8
 31|W|C||228.60|19.7600||18.7500||16.7400|||017073CD6
 30|C|7440-47-3|MG/KG||10.7230||P|32.3||L|2.1|||*|U|1.8||51.0200|||
 |01700CC13
 31|W|C||267.70|50.8900||51.3700||50.8000|||017095D18

 20|1|MAX123S|F|LSF||20596|MAX123|93|09|17|11|14|G|1.01|08|01730BE3C
 21||LOW|||S308233-03|93|09|14||93|08|24||8|01731CC90
 22|||200|1.00||91.5|01732D656
 30|C|7440-22-4|MG/KG||10.7212|F|10.82|P|99.1|||75|125||U|3.4|
 |49.54 00|||01733EBC7
 31|W|C||328.00|48.8400||49.2000||50.5900|||01734F8DC
 30|C|7429-90-5|MG/KG|NAR|6859.9253|F|0.00|||U|22.8||31698.0000|

```

| | 017350E27
31|W|C| |308.20|31578.0000| |31766.0000| |31750.0000| | | |017361CF1
30|C|7440-39-3| |MG/KG| |326.3539|F|432.83|P|70.3| | | |N|75|125| |U|1.0|
|1508.0000| | |017373339
31|W|C| |493.40|1524.0000| |1504.4000| |1495.6000| | | |017384171
30|C|7440-41-7| |MG/KG| |10.4290|F|10.82|P|93.5| | | |75|125| |U|0.4|
|48.1900| | |0173956E4
31|W|C| |313.00|48.1900| |48.2000| |48.2000| | | |0174063EB
30|C|7440-70-2| |MG/KG|NAR|775.1772|F|0.00| | | | | |U|35.7|B|3581.9000| |
|017417903
31|W|C| |317.90|3572.0000| |3586.4000| |3587.4000| | | |01742874B
30|C|7440-43-9| |MG/KG| |10.4290|F|10.82|P|96.4| | | |75|125| |U|1.5|
|48.1900| | |017439CC6
31|W|C| |226.50|47.5200| |48.5300| |48.5200| | | |01744A9DC
30|C|7440-48-4| |MG/KG| |109.8523|F|108.21|P|98.4| | | |75|125| |U|1.5|
|507.6000| | |01745BFF2
31|W|C| |228.60|505.2500| |508.2700| |509.2800| | | |01746CDA1
30|C|7440-47-3| |MG/KG| |52.0002|F|43.28|P|102.3| | | |75|125| |U|1.8|
|240.2800| | |01747E369
31|W|C| |267.70|239.3500| |240.2800| |241.2000| | | |01748F10C

20|1|MAX123L|F|LDF| |20596|MAX123|93|09|17|11|17| | |03|017696573
21| |LOW| | |S308233-04| | |93|08|24| | |017707255
22| | | | | |5.00| |91.5|017717B8D
30|C|7440-22-4| |UG/L|BDL|17.00| | | | | | |U|3.4|U|0.6100| | |017728DDF
31|W|C| |328.00|1.4500| |-0.3800| |0.7800| | | |017739A7B
30|C|7429-90-5| |UG/L| |25575.50| |P|14.5| | | | |E|U|22.8| |5115.1000| |
|01774AE69
31|W|C| |308.20|5038.6000| |5126.4000| |5180.3000| | | |01775BCAC
30|C|7440-39-3| |UG/L|LTC|111.30| |P|5.6| | | | |U|1.0|B|22.2600| |
|01776D0AA
31|W|C| |493.40|22.2600| |22.7700| |21.7500| | | |01777DDB9

```

2.0 GFAA

2.1 START OF A GFAA RUN WITH RECORD TYPES 10 & 16 AND THE FIRST THREE TYPE 20 RECORDS

```

10|93|09|22|11|38|F|ILM04.0|ABC|TESLAB| | |68-D2-0039|F2|
|TEST LABS INC.|3|000001860
16|93|09|22|16|07|Y| | |000011FFF

```

20|1|IDL|07|15|000044A4C
 30|C|7439-92-1|U|1.4|00005584F

20|1|LRV|09|22|0000665FC
 30|C|7439-92-1|000077317
 32|000087C1E
 34|283.30|3|100|000098447

20|1|BCD|09|22|0001091D2
 30|C|7439-92-1|000119EED
 35|BG|BS|00012A4CF

2.2 INSTRUMENT CALIBRATION STANDARDS BLANK (S0) & THREE OTHER STANDARDS

20|1|S0|1|20596|MAX123|93|09|22|11|38|00013B309
 21|0 PPB|TESLAB|00014BEA6
 22|1.00|00015C70D
 30|C|7439-92-1|T|0.0|U|1.4|U|0.0000|00016DA71
 31|W|B|283.30|0.0000|0.0000|00017E483

20|1|S3|1|20596|MAX123|93|09|22|11|42|00018F2BB
 21|3 PPB|TESLAB|00019FE5B
 22|1.00|0002006C2
 30|C|7439-92-1|T|3.0|U|1.4|0.0280|000211903
 31|W|B|283.30|0.0290|0.0270|000222318

20|1|S50|1|20596|MAX123|93|09|22|11|47|000233187
 21|50 PPB|TESLAB|000243D59
 22|1.00|0002545C0
 30|C|7439-92-1|T|50.0|U|1.4|0.2765|000265897
 31|W|B|283.30|0.2760|0.2770|0002762DE

20|1|S100|1|20596|MAX123|93|09|22|11|51|000287174
 21|100 PPB|TESLAB|000297D72
 22|1.00|0003085D9
 30|C|7439-92-1|T|100.0|U|1.4|0.5035|000319934
 31|W|B|283.30|0.5050|0.5020|00032A3A7

2.3 ANALYSIS OF A FIELD BLANK SAMPLE

SAMPLE & ITS ANALYTICAL SPIKE SAMPLE WITH QC CODE FRB

```
20|1|MAX124|1|FRB|20596|MAX123|93|09|22|12|58|ML|100|1|00092D8C5
21||LOW|||S308233-05|93|09|14|93|08|20||8|00093E714
22|||||||100|1.00||0.0|00094F0DE
92|COLORLESS|COLORLESS|CLEAR|CLEAR||00096FE00
30|C|7440-28-0|UG/L|BDL|3.0000|||||||U|3.0|U|0.4630|235.19|
|0009711EC
31|W|C||276.80|1.2330||-0.3070|||||000981D63
```

```
20|1|MAX124A|1|FRB|20596|MAX123|93|09|22|13|03|||1|000992CE7
21||LOW|||S308233-05|||93|08|20|||0010039C4
22|||||||1.00||0.0|0010142FC
30|C|7440-28-0|UG/L|20.9380|F|20.00|P|104.7||||85|115|U|3.0|
|20.9380|3.3700|001025A7B
31|W|C||276.80|21.4370|20.4390|||||001036635
```

2.4 SPIKE SAMPLE RECOVERY & DUPLICATES PERFORMED ON THE SAME SAMPLE (QC CODES LSO & LSF, AND LD1 & LD2)

NOTE: SAMPLE MAX123A CAN HAVE EITHER QC CODE LSO OR LD1

```
20|1|MAX123|F|LSO|20596|MAX123|93|09|22|12|40|G|1.00|1|00094E6D2
21||LOW|||S308233-01|93|09|14|93|08|24||8|00095F526
22|||||||200|1.00||91.5|00096FEEC
30|C|7782-49-2|MG/KG|BDL|0.8087|||||||U|3.7|U|1.5305|161.38|
|000991F6C
31|W|C||196.00|3.2770||-0.2160|||||001002AE5
```

```
20|1|MAX123|F|LD1|20596|MAX123|93|09|22|12|40|G|1.00|1|001013BFE
21||LOW|||S308233-01|93|09|14|93|08|24||8|001024A52
22|||||||200|1.00||91.5|001035418
30|C|7782-49-2|MG/KG|BDL|0.8087|||||||U|3.7|U|1.5305|161.38|
|001067498
31|W|C||196.00|3.2770||-0.2160|||||001002AE5
```

```
20|1|MAX123A|F|LD1|20596|MAX123|93|09|22|12|45|||1|001089061
21||LOW|||S308233-01|||93|08|24|||001099D43
22|||||||1.00||91.5|00110A677
30|C|7782-49-2|UG/L|10.2050|F|10.00|P|102.0||||85|115|U|3.7|
|10.2050|8.1000|00111BE28
31|W|C||196.00|10.7890|9.6210|||||00112C9B2
```

20|1|MAX123D|F|LD2||20596|MAX123|93|09|22|12|50||G|1.00|1|001479A0E
 21||LOW|||S308233-02|93|09|14||93|08|24|||8|00148A862
 22|||||||200|1.00||90.9|00149B22B
 30|C|7782-49-2|MG/KG|LTC|0.8509||P|200.0|||||||U|3.7|B|3.8930|
 |1.0172||00150C661
 31|W|C||196.00|3.9210||3.8650||||||00151D1F9

20|1|MAX123DA|F|LD2||20596|MAX123|93|09|22|12|55||||1|00152E28C
 21||LOW|||S308233-02||||93|08|24||||00153EF6E
 22|||||||1.00||91.5|00154F8A2
 30|C|7782-49-2|UG/L||13.5660|F|10.00|P|96.7|||||85|115||U|3.7||13.566|
 |1.0320||001550FDF
 31|W|C||196.00|13.6650||13.4670||||||001561B3F

20|1|MAX123S|F|LSF||20596|MAX123|93|09|22|13|00||G|1.01|1|001572CC1
 21||LOW|||S308233-03|93|09|14||03|08|24|||8|001583B15
 22|||||||200|1.00||91.5|0015944DB
 30|C|7782-49-2|MG/KG||1.9178|F|2.16|P|88.8|||||75|125||U|3.7||8.8615|
 |3.2000||001605B08
 31|W|C||196.00|8.6610||9.0620||||||001616692

2.5 DUPLICATES, WITH THE RESULT OF THE DUPLICATE SAMPLE QUANTITATED BY THE MSA (QC CODES LD1, LD2, MS0, MS1, MS2, MS3)

NOTE: WHEN THE RESULT OF THE DUPLICATE SAMPLE IS QUANTITATED BY THE MSA, THE DATA FOR THE RPD, THE CONTROL LIMIT(CRDL), AND THE * QC LIMIT QUALIFIER ARE ENTERED ON THE MS0 TYPE 30 RECORD. THAT IS, DATA FOR THE DUPLICATE ANALYSIS THAT ARE ENTERED ON FORM VI MUST BE ENTERED ON THE TYPE 30 RECORD OF THE EPA SAMPLE NUMBER THAT HAS THE 'D0' SUFFIX (E.G., MAX123D0)

20|1|MAX123|F|LD1||20596|MAX123|93|09|22|12|40||G|1.00|1|00135FD7E
 21||LOW|||S308233-01|93|09|14||93|08|24|||8|001360BD2
 22|||||||200|1.00||91.5|001371598
 30|C|7440-38-2|MG/KG||4.7259|||||||U|2.7||21.6210||0.0000|
 |0014034CA
 31|W|C||197.20|21.6210||21.6210||||||001414075

20|1|MAX123A|F|LD1||20596|MAX123|93|09|22|12|45||||1|0014250C2
 21||LOW|||S308233-01||||93|08|24||||001435DA4
 22|||||||1.00||91.5|0014466D8
 30|C|7440-38-2|UG/L||44.2020|F|20.00|P|112.9|||||85|115||U|2.7|
 |44.2020||2.7700||001457E51
 31|W|C||197.20|45.0690||43.3350||||||001468A0E

20|1|MAX123D|F|LD2||20596|MAX123|93|09|22|12|50||G|1.00|1|001479B61
 21||LOW|||S308233-02|93|09|14||93|08|24|||8|00148A9B5
 22|||200|1.00||91.5|00149B37E
 30|C|7440-38-2||MG/KG|RIN|5.8114|||U|2.7||26.5870||1.5100|
 |00150C77C
 31|W|C||197.20|26.8700||26.3040|||00151D335

20|1|MAX123DA|F|LD2||20596|MAX123|93|09|22|12|55|||1|00152E3C8
 21||LOW|||S308233-02|||93|08|24|||00153F0AA
 22|||1.00||91.5|00154F9DE
 30|C|7440-38-2||UG/L|RIN|49.9655|F|20.00|P|116.9|||85|115||U|2.7|
 |49.9655||2.6500||001551279
 31|W|C||197.20|49.0290||50.9020|||001561E34

20|1|MAX123D0|F|MS0||20596|MAX123|93|09|22|13|00||G|1.00|1|00270330F
 21||LOW|||S308233-02|93|09|14||93|08|24|||8|002714163
 22|||200|2.50||91.5|002724B2A
 30|C|7440-38-2||MG/KG||7.8142|F|0.0|P|49.3|C|0.9958|L|2.2|||*|U|2.7|
 |14.3|||002735F59
 31|W|B||197.20|0.0550|||002746977

20|1|MAX123D1|F|MS1||20596|MAX123|93|09|22|13|03||G|1.00|1|002757B10
 21||LOW|||S308233-02|93|09|14||93|08|24|||8|002768964
 22|||200|2.50||91.5|00277932B
 30|C|7440-38-2|||F|10.0|||U|2.7|||00278A230
 31|W|B||197.20|0.0810|||00279AC4D

20|1|MAX123D2|F|MS2||20596|MAX123|93|09|22|13|06||G|1.00|1|00280BDEB
 21||LOW|||S308233-02|93|09|14||93|08|24|||8|00281CC3F
 22|||200|2.50||91.5|00282D606
 30|C|7440-38-2|||F|20.0|||U|2.7|||00278A230
 31|W|B||197.20|0.1240|||00284EF27

20|1|MAX123D3|F|MS3||20596|MAX123|93|09|22|13|09||G|1.00|1|0028500C1
 21||LOW|||S308233-02|93|09|14||93|08|24|||8|002860F15
 22|||200|2.50||91.5|0028718DC
 30|C|7440-38-2|||F|30.0|||U|2.7|||00278A230
 31|W|B||197.20|0.1600|||0028931FE

3.0 MERCURY (CVAA OR AVAA)

3.1 START OF A MERCURY RUN WITH RECORD TYPES 10 & 16 AND THE FIRST TWO TYPE 20 RECORDS

10|93|09|09|08|44|CV|ILM04.0|ABC|TESLAB|||68-D2-0039|M3|
|TEST LABS INC.|16|0000018F7
16|93|09|09|14|34|N|||000012099

20|1|IDL|||93|07|15|||1|000044AEB
30|C|7439-97-6|||U|0.1|||0000558F4

20|1|LRV|||93|09|09|||1|0000666A6
30|C|7439-97-6|||0000773CB
32|||000087D02
34|||253.70|0.2|5|||00009852D

3.2 MERCURY INSTRUMENT CALIBRATION STANDARDS BLANK (S0) AND FOUR OTHER STANDARDS

20|1|S0|1||20596|MAX123|93|09|09|08|44|||1|00010936F
21|||0 PPB|||TESLAB||000119F0C
22|||1.00|||00012A773
30|C|7439-97-6|||T|0.0|||U|0.1|U|0.0122|||00013BAD9
31|W|C||253.70|0.0122|||00014C4EC

20|1|S0.2|1||20596|MAX123|93|09|09|08|48|||1|00015D392
21|||0.2 PPB|||TESLAB||00016DF8F
22|||1.00|||00017E7F6
30|C|7439-97-6|||T|0.2|||U|0.1|B|0.1987|||00018FB5E
31|W|C||253.70|0.1987|||000190571

20|1|S1.0|1||20596|MAX123|93|09|09|08|53|||1|000201412
21|||1.0 PPB|||TESLAB||00021200E
22|||1.00|||000222875
30|C|7439-97-6|||T|1.0|||U|0.1|1.0128|||000233BDC
31|W|C||253.70|1.0128|||0002445EF

20|1|S2.0|1||20596|MAX123|93|09|09|08|57|||1|000255495
21|||2.0 PPB|||TESLAB||000266092
22|||1.00|||0002768F9
30|C|7439-97-6|||T|2.0|||U|0.1|2.0055|||000287C61
31|W|C||253.70|2.0055|||000298674

20|1|S5.0|1|||20596|MAX123|93|09|09|09|01|||1|000309513
 21|||5.0 PPB|||TESLAB||00031A113
 22|||1.00|||00032A97A
 30|C|7439-97-6|||T|5.0|||U|0.1||4.9952|||00033BCE5
 31|W|C|253.70|4.9952|||00034C6F8

3.3 SPIKE SAMPLE RECOVERY & DUPLICATES PERFORMED ON DIFFERENT SAMPLES (QC CODES LSO & LSF, AND LD1 & LD2)

20|1|MAX123|F|LSO||20596|MAX123|93|09|09|13|20||G|0.20|1|002106798
 21||LOW|||S308233-01|93|09|08||93|08|24|||8|0021175EF
 22|||100|1.00||91.5|002127FB4
 30|C|7439-97-6||MG/KG|BDL|0.0546|||U|0.1|U|0.0349|||002159EC0
 31|W|C|253.70|0.0349|||00216A8E3

20|1|MAX123S|F|LSF||20596|MAX123|93|09|09|13|25||G|0.20|1|00229534B
 21||LOW|||S308233-03|93|09|08||93|08|24|||8|0023061A2
 22|||100|1.00||91.5|002316B67
 30|C|7439-97-6||MG/KG||0.5664|F|0.55|P|103.0|||75|125||U|0.1||1.0366|
 ||00232807A
 31|W|C|253.70|1.0366|||002338A9D

20|1|MAX126|F|LD1||20596|MAX123|93|09|09|13|30||G|0.22|1|00217B9F5
 21||LOW|||S308233-06|93|09|08||93|08|24|||8|00218C84C
 22|||100|1.00||85.6|00219D211
 30|C|7439-97-6||MG/KG||1.3685|||U|0.1||2.5771|||00222F11D
 31|W|C|253.70|2.5771|||00223FB40

20|1|MAX126D|F|LD2||20596|MAX123|93|09|09|13|35||G|0.21|1|002240C9D
 21||LOW|||S308233-07|93|09|08||93|08|24|||8|002251AF4
 22|||100|1.00||85.1|0022624BC
 30|C|7439-97-6||MG/KG|BDL|0.0556||P|200.0||L|0.11|||*|U|0.1|U|0.0278|
 ||002273795
 31|W|C|253.70|0.0278|||0022841B9

3.4 SPIKE SAMPLE RECOVERY & DUPLICATES PERFORMED ON THE SAME SAMPLE (QC CODES LSO & LSF, AND LD1 & LD2)

20|1|MAX126|F|LSO||20596|MAX123|93|09|09|16|10||G|0.20|1|002106798
 21||LOW|||S308233-06|93|09|08||93|08|24|||8|0021175EF
 22|||100|1.00||91.5|002127FB4
 30|C|7439-97-6||MG/KG||0.6429|||U|0.1||1.1765|||002159EC0
 31|W|C|253.70|1.1765|||00216A8E3

20|1|MAX126|F|LD1||20596|MAX123|93|09|09|16|10||G|0.20|1|00217B9F5
 21||LOW|||S308233-06|93|09|08||93|08|24|||8|00218C84C
 22|||||||100|1.00|||91.5|00219D211
 30|C|7439-97-6|MG/KG||0.6429|||||||U|0.1||1.1765|||002159EC0
 31|W|C||253.70|1.1765|||||||00223FB40

20|1|MAX126D|F|LD2||20596|MAX123|93|09|09|16|15||G|0.21|1|002240C9D
 21||LOW|||S308233-07|93|09|08||93|08|24|||8|002251AF4
 22|||||||100|1.00|||90.9|0022624BC
 30|C|7439-97-6|MG/KG||0.2231||P|97.0||L|0.11|||*|U|0.1||0.4286|||
 |002273795
 31|W|C||253.70|0.4286|||||||0022841B9

20|1|MAX126S|F|LSF||20596|MAX123|93|09|09|16|20||G|0.20|1|00229534B
 21||LOW|||S308233-08|93|09|08||93|08|24|||8|0023061A2
 22|||||||100|1.00|||91.5|002316B67
 30|C|7439-97-6|MG/KG||0.9710|F|0.55|P|59.7|||N|75|125|U|0.1||1.7769|
 ||00232807A
 31|W|C||253.70|1.7769|||||||002338A9D

3.5 INITIAL CALIBRATION VERIFICATION (ICV) WITH LVM QC CODE

20|1|ICV|1|LVM||20596|MAX123|93|09|09|09|06|||1|00035D687
 21|||||ICV-5|||||||ICF(0791)||00036E25E
 22|||||||2.00|||00037EAC6
 30|C|7439-97-6|UG/L||4.91|T|4.9|P|100.2|||||80.0|120.0|U|0.1||2.4559|
 ||00038FFD0
 31|W|C||253.70|2.4559|||||||0003909FC

3.6 LABORATORY CONTROL SAMPLE (SOLID) WITH LCM QC CODE

20|1|LCSS|F|LCM||20596|MAX123|93|09|09|12|24||G|0.20|1|001256DBA
 21|||||LCSHG|93|09|08|||QAL-0287||8|001267B1B
 22|||||||100|10.00|||001278443
 30|C|7439-97-6|MG/KG||13.9|T|12.7|P|109.4|||||8.5|17.0|U|0.1||2.7719|
 ||00128996D
 31|W|C||253.70|2.7719|||||||00129A39A

4.0 CYANIDE (CA, AS, C, T)

4.1 START OF A CYANIDE RUN WITH RECORD TYPES 10 & 16 AND THE FIRST TWO TYPE 20 RECORDS

10|93|09|01|14|09|CA|ILM04.0|ABC|TESLAB|||68-D2-0039|C1|
|TEST LABS INC.|7|00000189C
16|93|09|01|15|03|Y|||000012033

20|1|IDL|||91|10|15|||1|000044A74
30|I|||U|10.0|||0000556DC

20|1|LRV|||93|09|01|||1|000066486
30|I|||000076FDA
32|||IT|45.00||000087917
34|||620.00|10|400|||000098169

4.2 CYANIDE INSTRUMENT CALIBRATION STANDARDS BLANK (S0) AND FIVE OTHER STANDARDS

20|1|S0|1||20596|MAX123|93|09|01|14|09|||1|000108FA1
21|||0 PPB|||TESLAB||000119B3E
22|||1.00||00012A3A5
30|I|||T|0.0|||U|10.0|U|0.3543|||00013B48B
31|W|C||620.00|0.3543|||00014BD34

20|1|S10|1||20596|MAX123|93|09|01|14|10|||1|00015CB95
21|||10 PPB|||TESLAB||00016D763
22|||1.00||00017DFCA
30|I|||T|10.0|||U|10.0||11.1700|||00018F0D2
31|W|C||620.00|11.1700|||00019F97B

20|1|S40|1||20596|MAX123|93|09|01|14|11|||1|0002007E0
21|||40 PPB|||TESLAB||0002113B1
22|||1.00||000221C18
30|I|||T|40.0|||U|10.0||38.4000|||000232D23
31|W|C||620.00|38.4000|||0002435CC

20|1|S100|1||20596|MAX123|93|09|01|14|12|||1|00025445F
21|||100 PPB|||TESLAB||00026505D
22|||1.00||0002758C4
30|I|||T|100.0|||U|10.0||99.7400|||000232D23
31|W|C||620.00|99.7400|||0002972A5

20|1|S200|1|||20596|MAX123|93|09|01|14|12|||1|000308139
 21|||||200 PPB|||||||TESLAB|||000318D38
 22|||||||||1.00|||00032959F
 30|I|||||T|200.0|||||||U|10.0|201.3000|||00033A6D8
 31|W|C||620.00|201.3000|||||||00034AF81

20|1|S400|1|||20596|MAX123|93|09|01|14|13|||1|00035BE18
 21|||||400 PPB|||||||TESLAB|||00036CA19
 22|||||||||1.00|||00037D280
 30|I|||||T|400.0|||||||U|10.0|399.5000|||00038E3BB
 31|W|C||620.00|399.5000|||||||00039EC64

4.3 PREPARATION BLANK (SOIL) WITH LRB QC CODE

20|1|PBS|F|LRB||20596|MAX123|93|09|01|14|23||G|1.00|1|000928FA0
 21|||||PB|93|08|30||||||8|000939A40
 22|||||||||50|1.00|||00094A30C
 30|I|||MG/KG|BDL|0.500|||||||U|10.0|U|-0.1130|||00095B433
 31|W|C||620.00|-0.1130|||||||00096BE6F

4.4 LABORATORY CONTROL SAMPLE (SOIL) WITH LCM QC CODE

20|1|LCSS|F|LCM||20596|MAX123|93|09|01|14|24||G|1.00|1|00097CF4D
 21|||||LCSCN|93|08|30|||||QAL-0689||8|00098DCB0
 22|||||||||50|1.00|||00099E57C
 30|I|||MG/KG||5.0|T|5.6|P|89.3|||||4.3|6.9|U|10.0|100.0933|||
 |00100F89B
 31|W|C||620.00|100.0933|||||||001010315

4.5 CONTINUING CALIBRATION VERIFICATION (CCV) WITH LVC QC CODE

20|1|CCV|1|LVC||20596|MAX123|93|09|01|14|30|||1|0015045A3
 21|||||200 PPB|||||||TESLAB|||0015151A2
 22|||||||||1.00|||001525A09
 30|I|||UG/L||188.48|T|200.0|P|94.2|||||85.0|115.0|U|10.0|188.4772|||
 |001536E87
 31|W|C||620.00|188.4772|||||||001547916

4.6 SPIKE SAMPLE RECOVERY & DUPLICATES PERFORMED ON THE SAME SAMPLE (QC CODES LSO & LSF, AND LD1 & LD2)

20|1|MAX123|F|LSO||20596|MAX123|93|09|01|14|35||G|1.07|1|001955D8E
 21||LOW|||S308233-01|93|08|30|93|08|24|||8|001966BDF
 22|||||||||50|1.00|||91.5|001977578

30|I||MG/KG|BDL|0.5107|||U|10.0|U|-0.3521|||002009309
31|W|C||620.00|-0.3521|||002019D4B

20|1|MAX123|F|LD1||20596|MAX123|93|09|01|14|35||G|1.07|1|00202AE62
21||LOW|||S308233-01|93|08|30|93|08|24||8|00203BCB3
22|||50|1.00||91.5|00204C64C
30|I||MG/KG|BDL|0.5107|||U|10.0|U|-0.3521|||00207E3DD
31|W|C||620.00|-0.3521|||00208EE1F

20|1|MAX123D|F|LD2||20596|MAX123|93|09|01|14|36||G|1.05|1|00209FF7A
21||LOW|||S308233-02|93|08|30|93|08|24||8|002100DCB
22|||50|1.00||90.9|002111767
30|I||MG/KG|BDL|0.5204|||U|10.0|U|-0.6395|||0021228D6
31|W|C||620.00|-0.6395|||002133324

20|1|MAX123S|F|LSF||20596|MAX123|93|09|01|14|37||G|1.01|1|0021444AD
21||LOW|||S308233-03|93|08|30|93|08|24||8|0021552FE
22|||50|2.00||91.5|002165C98
30|I||MG/KG||25.8410|F|27.05|P|95.5|||75|125|U|10.0||238.8096|||
|0021770C0
31|W|C||620.00|238.8096|||002187B4E